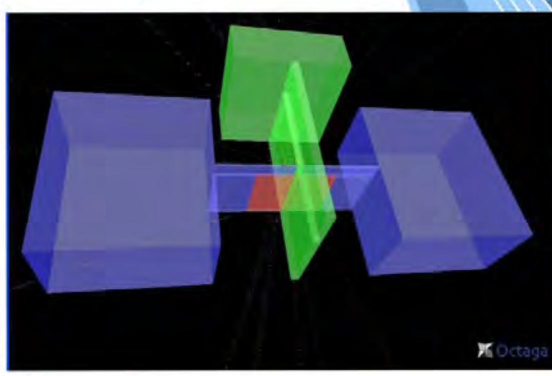
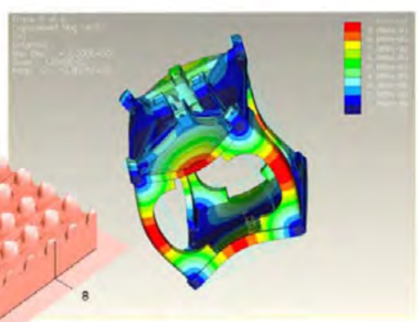
Manufacturing Engineering Laboratory

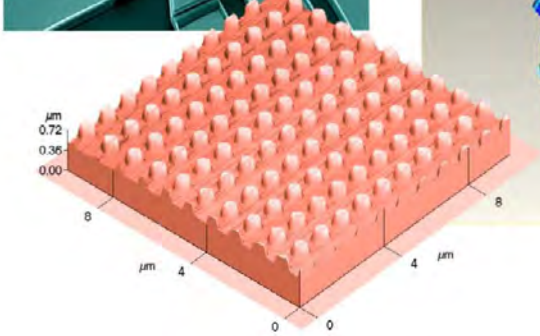
Precision Engineering

2009

Program Technical Accomplishments







NIST
National Institute of
Standards and Technology
U.S. Department of Commerce

PRECISION ENGINEERING DIVISION

Program Technical Accomplishments 2009

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December 2009

Next-Generation Nanometrology Program

Nanomanufacturing Metrology Program

Dimensional Metrology Program

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Beamie Young is acknowledged and thanked for her assistance for her excellent initial "InDesign" basic training and editing the final layout of this document.SEMATECH and the Office of Microelectronics Programs are also acknowledged and thanked for their partial support of many of the projects.

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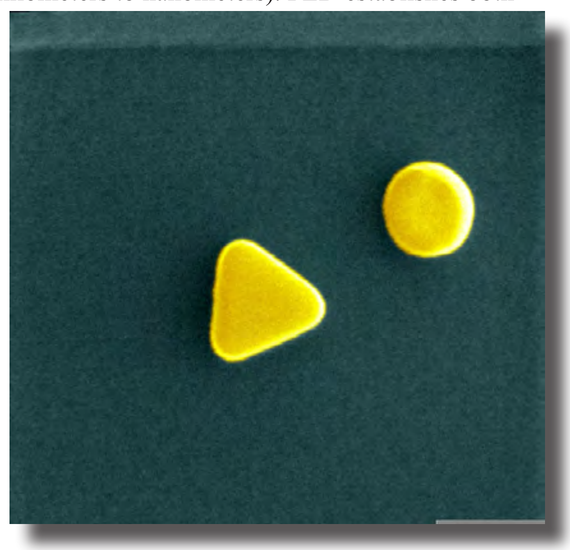
Contents

Precision Engineering Division

The Precision Engineering Division (PED) is one of five divisions of the Manufacturing Engineering Laboratory (MEL) at the National Institute of Standards and Technology (NIST). PED provides the United States with the foundation for dimensional measurements ranging over 12 orders of magnitude (from kilometers to nanometers). PED establishes both artifact and documentary standards through research, development, measurement services, and information dissemination. PED also conducts infrastructural research and development in precision-engineered length-metrology-intensive systems in instrumentation for both measurement and production.



Dimensional metrology of 60 nm gold nanoparticles

PED delivers to industry important length-related measurements, standards, and technology services that directly support U.S. manufacturing's products and processes. These standards are measured by a whole array of instrumentation ranging from atomic force microscopes to conventional and frameless coordinate measuring machines (CMM) using contact, particle beam and optical measuring probes, machines, and systems. Features of interest range in size from one kilometer (calibrated telecommunication cables) to meters (laser trackers) to nanometers (CD metrology instruments and nano-particle dimensional metrology).

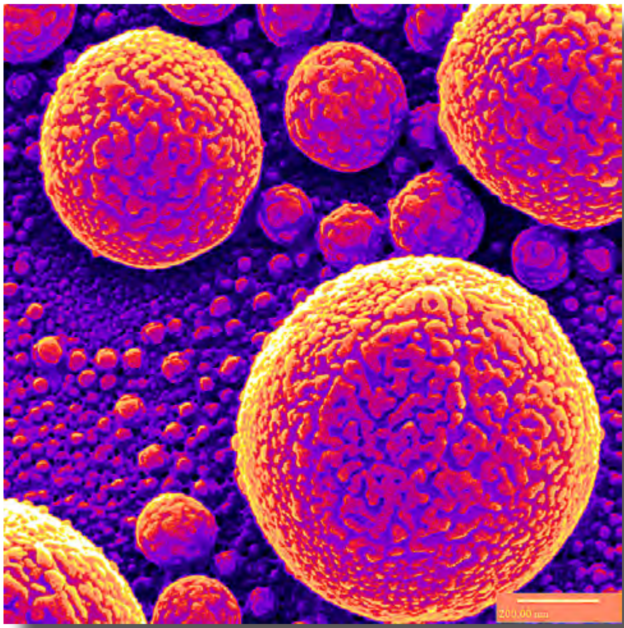
Traceable linescale metrology

PED is committed to developing the metrology needed to support U. S. manufacturing and has been instrumental in the development of instrumentation, metrology and standards especially for the semiconductor, automotive and aerospace industries. PED is also a primary partner in the development of the needed metrology for the Department of Defense and many of the other agencies. The continual push of the semiconductor industry to smaller and smaller gate structures has also pushed PED to ever improving nanoscale measurements and standards and a drive to encourage instrument manufacturers for higher performance. This has strategically positioned PED for leadership in the development of accurate metrology for nanotechnology and nanomanufacturing. Thus, for over a decade, the Manufacturing Engineering Laboratory and the Precision Engineering Division have been deeply involved in the development of metrology for the nascent nanomanufacturing industry (Lyons and Postek, 2009).

The spectrum of metrology instrumentation within PED includes optical, particle beam, mechanical, electrical, and quantum-mechanical phenomena, all referred to the International System of Units (SI) unit of length (modernized metric system), which is defined through the use of stabilized lasers and displacement interferometry.

PED has developed three Manufacturing Engineering Laboratory-level programs to best utilize the talents of its personnel and thus meet its mission with maximum effectiveness. These three programs outlined in this document and the projects described. These programs which are described in this publication are:

- **Dimensional Metrology**
- **Nanomanufacturing Metrology**
- **Next-Generation Nanometrology**

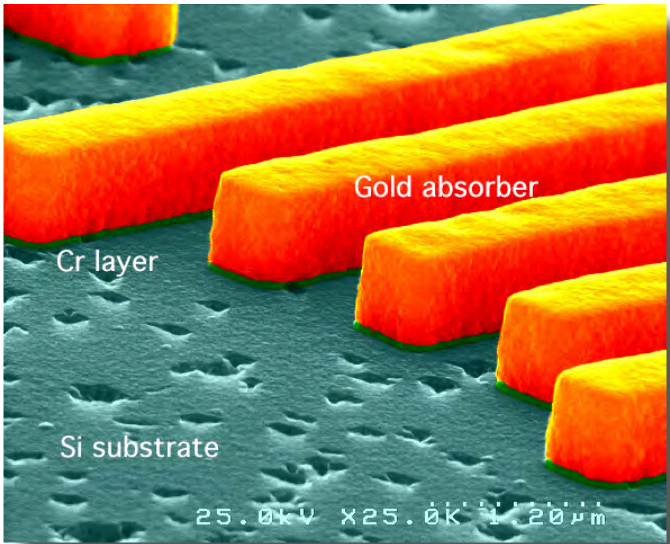


Dimensional metrology for nanomanufacturing

65 meter long laser scanner test range

Reference:

Lyons, K., and Postek, M. T. 2009. Historic commitment of the NIST Manufacturing Engineering Laboratory to nanomanufacturing and nanometrology. SPIE Proceedings. Proc. SPIE 7405 740503-1 – 740503-18.



Semiconductor linewidth metrology

PED Mission:

The mission of PED is to provide the foundation of dimensional measurement that meets the needs of the U.S. industrial and scientific communities by:

- Conducting research in dimensional measurements;
- Developing new measurement methods;
- Providing measurement services;
- Developing National and International artifact and documentary standards; and
- Disseminating the resulting technology and length-based standards.

Also within the mission of PED, is to provide dimensional metrology assistance to other federal government agencies in order to address problems and needs that leverage NIST expertise, facilities, and capabilities.

The mission of PED reflects its responsibility to perform four core functions: research, development, measurement services, and information dissemination. Each core function includes a variety of processes which serve numerous customers, collectively identified as industrial and scientific communities, both public and private. PED values accuracy, responsiveness, objectivity, value, knowledge, expertise, state of the art, integrity, and honesty in the performance of this mission.

PED Vision:

The vision of PED is to conduct research and development in precision-engineered length-metrology-intensive systems in both measuring and production machines.

The Precision Rngineering Division delivers to industry important length-related measurements, standards, and technology services that support U.S. manufacturing's products and processes. Features of interest range in size from multiple meters to sub-nanometer. These are measured by framed and frameless general special-purpose measuring probes, machines, and systems. The spectrum includes optical, mechanical, electrical, and quantum-mechanical phenomena using the first-principle of the Systeme Internationale (SI) unit of length (modernized metric system) by means of stabilized lasers and displacement interferometry.

Metrology for micromachining

PED is broken up into four organizational units: Large-Scale Coordinate Metrology Group (821.11), Engineering Metrology Group (821.12), Surface and Microform Metrology Group (821.13) and the Nanoscale Metrology Group (821.14).

- **Large-Scale Coordinate Metrology Group (821.11)** supports the Division's mission with a concentration on measurements of one meter or larger. For example, measurements are made by coordinate measuring machines and frameless coordinate length-metrology systems. These involve mechanical-probe, laser-ranging, theodolite, and related interferometric systems.
- **Engineering Metrology Group (821.12**) supports the Division's mission with a concentration on measurements of one millimeter to one meter. These measurements are often of complex shape, such as turbine blades, threaded fasteners, and gears. Measurements are made by general and special-purpose feature and coordinate measuring systems.
- **Surface and Microform Metrology Group (821.13)** supports the division's mission with a concentration on measurements of one micrometer to one millimeter where surface roughness and microform are critical. Measurements are made by tunneling microscopes, mechanical profilometry, phase-measuring interferometry and related techniques.
- **Nanoscale Metrology Group (821.14)** supports the division's mission with a concentration on measurements of one nanometer to one micrometer. Measurements are made by tunneling, atomic-force, electron, and visible- and ultraviolet-light microscopies. The group places special emphasis on satisfying the advanced needs of U.S. microelectronic manufacturing industries.

Dimensional Metrology Program -

Realizing and Disseminating the Meter

Program Manager: Dr. Steven Phillips

Challenge:

To promote innovation and enhance U. S. productivity by optimizing MEL's dimensional metrology portfolio to take advantage of advances in information, computational, and optoelectronic measurement technology for high accuracy dimensional metrology.

Overview:

The Dimensional Metrology Program (DMP) addresses selected needs in dimensional metrology over length scales ranging from micrometers to kilometers. This includes calibrations of measuring instruments such as laser interferometers and laser trackers, a wide array of engineering gauges, producing standard reference materials, and performing specialized measurements, e.g., on high accuracy coordinate measuring machines (CMMs). The program also provides expertise and representation of U. S. interests in numerous national and international standards committees.

The DMP focuses on developing dimensional measurement infrastructure to support significant improvements in U.S. productivity. In particular, the DMP targets building new measurement capability for enabling technologies and high value products in which the U.S. has a significant industrial presence. Selected national and international standards, particularly in emerging technologies, are supported to promote international trade, foster innovation, and reduce manufacturing costs. The program also strives to increase the leverage of NIST's measurements through deep penetration of its measurements into the U.S. metrology chain.

Strategically, the DMP develops unique measurement capabilities – typically for measurements with a combination of extremely low uncertainty and complex measurands – that are difficult for other laboratories to replicate; this differentiates the DMP from other top tier calibration laboratories and from universities. The DMP implements its objectives through a series of objectives, each containing a set of time staggered projects. The objectives are forward looking that will create and deliver solutions to significant dimensional metrology problems, typically leading to new measurement services.

NIST has the unique mission to work with industry to foster innovation and trade that promotes U. S. competitiveness. NIST's position is distinct from that of industry and universities. The

DMP selects projects that target NIST's mission and cannot effectively be addressed by other constituencies. The selection of a metrology project balances several competing forces. Typically, measurement problems involve measurands of extreme technical difficulty and accuracy where the cost of measurements excludes private sector enterprises. Simultaneously, the program creates positive return on investment by targeting projects with significant leverage and value with net positive aggregate benefits to the nation. Additionally, the program recognizes the lower cost structure of universities and avoids projects that could be addressed more economically and efficiently by these institutions. In particular, the DMP exploits *economies of experience* and scope from NIST's unique long term institutional knowledge and capability which is unavailable to academia.

NIST's specific role as a neutral third party with technical excellence provides the DMP with a unique role for developing national and international standards that foster trade, create a level playing field, and promote emerging or high value technologies where the US has a significant presence. Standards have enormous leverage because they can formulate how an entire industry specifies and tests its products. Well written standards have positive externalities to industry by: avoiding costs of individualized (i.e. idiosyncratic) testing, optimizing capital purchases by allowing meaningful comparisons between different products (purchasing the right tool for the job), and lowering barriers to adopting new technology through the increased confidence and information imparted to industrial users which enhances early adoption of technology.

The Dimensional Metrology Program is composed of 6 projects:

- Ongoing Reimbursable Dimensional Calibrations and Quality System
- Improvements in Calibrations and Uncertainty Evaluation
- Optical Comb and Refractometry
- MicroFeature Calibration Development
- Complex Geometry Instrumentation and Standards
- Micrometer level surface finish metrology

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Ongoing Reimbursable Dimensional Calibrations and Quality System

Industrial Need:

US industries that involve dimensional metrology rely on NIST to provide state of the art high accuracy calibrations in order to maintain the measurement traceability and international competitiveness. To assure the international acceptance of NIST measurements, NIST has developed a Quality System based on the ISO 17025 Standard.

Project Objective:

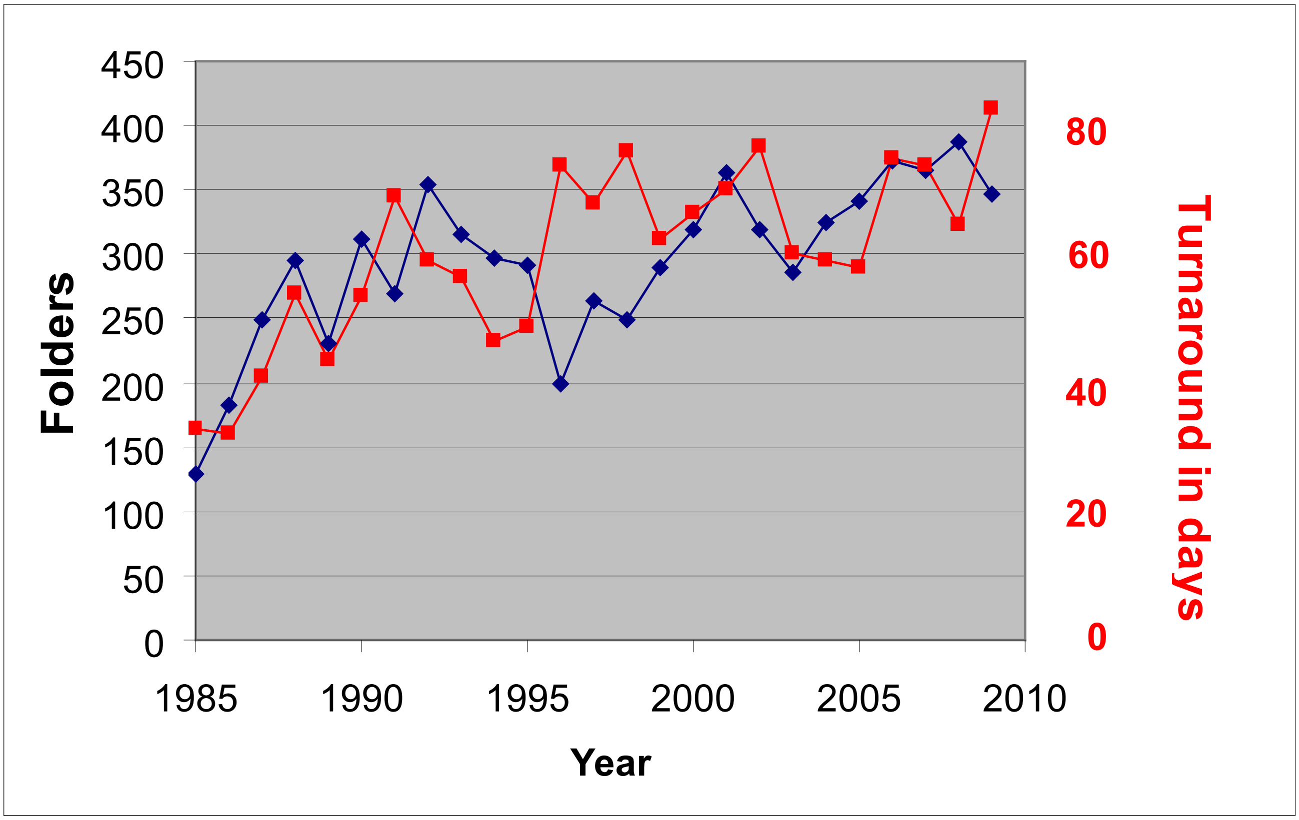
To provide industry high accuracy dimensional measurements within an internationally accepted quality system. DMP measurements are targeted to provide measurement results that are either of very high value to our customers or high leverage into the industrial metrology community, i.e. a single calibration may propagate into hundreds or thousands of subsequent measurement results.

Technical Approach:

The basic approach for all of our calibration development is to buy high precision, high repeatability instruments and then with a combination of advanced measurement methods and decades of metrological experience and data, improve the results obtained from the instruments. The methods include error mapping, improved thermal control, redundant measurements, reversal techniques, and statistical process control.

The program also seeks to cull out measurement services in technologically declining areas. For example, level rod calibrations (used in civil engineering) have been eliminated since GPS offers higher accuracy over large distances. The calibration of mechanical sieves was eliminated in FY-08 as sieves are a relatively low-value calibration that can be performed by secondary calibration laboratories.

Quality assurance metrics have been implemented and have continuously improved over the past three years. On time delivery is now over 97%, a significant increase from less than 60% only five years ago. However, turnaround time has remains problematic as the number of customers has significantly risen but the cost per customer has declined, spreading the fixed staff over a larger number of calibration activities; additional emphasis is being focused on reducing turnaround time.



The PED Quality System had its second audit by internal assessors with the guidance of NVLAP. The Quality System passed with only minor issues and these findings were addressed. The audit was accepted by the Assessment Review Board this summer. There were no major changes in the quality system.

The most important approach for excellent calibrations is to hire and develop excellent metrologists. The addition of Chris Blackburn (B.S. in mathematics), Massi Ferrucci (B.S. in physics) and Michale Braine (A.S. in metrology) and Alan Zheng (B.S. Mechanical Engineering) has been an important step towards making the staff more flexible in a time where our largest increases in workload are for Special Tests. Unfortunately the number of full time equivalent staff members has been about the same for a long time.

The graph above shows the workload, by number of calibrations estimated by the number of jobs (folders) each year and the turnaround in days. The last point for FY 2009 is probably not its final value, the administrative time involved at end of year makes the system lag reality for a number of weeks. While the year to year changes are large, the basic trend is increasing workload leads to increasing turnaround.

Impact/Benefits:

The DMP's measurement service delivers, on an ongoing basis, high accuracy calibrations that focus on either high value components or high leverage calibrations. For example, the NIST tape calibrations have tremendous market penetration. US manufactures produce millions of measuring tapes each year that have accuracy and traceability from master tapes calibrated at NIST. Similarly, each NIST gauge block calibration explicitly controls – as cited by calibration report numbers – a thousand subsequent gauge block calibrations performed in industrial laboratories. The new, more flexible capability of the M48 has brought in a number of new customers including: The calibration of step gages (CMM manufacturers and service providers), ring gages, industry long range 1D instruments, and nozzles (NIST and industry), cylinders for the Calculable Capacitor (NIST), alignment fixtures for the James Webb Telescope (NASA) and a triple retroreflector for the SIMlite Astrometric Observatory (NASA).

PED has been working closely with the Butler County Community College metrology program to expose its students the ISO 17025 standard. Nearly all calibrations labs are now accredited and new metrologists should have some knowledge of the regulatory environment they will work in. After lectures and exercises, two metrology faculty

......each NIST gauge block calibration explicitly controls – as cited by calibration report numbers – a thousand subsequent gauge block calibrations performed in industrial laboratories.

and 5 of their students participated in the PED internal technical audit. The experience was very successful and we expect to repeat it on an annual basis.

Accomplishments:

- NIST Internal Assessment and approval of the Corrective Actions.
- PED NIST Assessment Approval from ARB.
- PED Internal Assessment and Management Review.
- Steadily increasing number of new customers
- Quality Metric mets: On-time delivery goal: 98%.
- M48 High Accuracy CMM begins calibration of ring gages in place of manual system.
- Roundness Calibrations taken over by new Mitutoyo HR-4000.
- Began regular calibrations on submillimeter feature gages using M48 and MTI UMAP.

Publications List

- Doiron, T. D., "Gauge Blocks - A Zombie Technology", NIST Journal of Research, Volume 113/3, pp. 175-184, 2008.

Presentations:

- Doiron, T, Eric Stanfield, Bryon Faust, John Stoup, Mary Abbott, "New Capabilities at NIST In Dimensional Metrology", NCSLI Symposium and Workshop, 2005.
- Stoup, J. R., Faust, B. S., Doiron, T. D., "Early Results From the NIST M48 CMM in the New AML Facility", Proceedings: Measurement Science Conference, Anaheim, CA, March 02-03, 2006.
- Song, J. , Low, S. R., Zheng, A., "Calibration Reproducibility Test for NIST's No. 3581 Standard Rockwell Diamond Indenter ", Proceedings of XVIII IMEKO World Congress, September 17-22, 2006.
- Doiron, T., "The Gage Block Seminar", two day seminar for industrial metrologists, held in conjunction with the Manufacturing & Measurement Conference & Workshop sponsored by Quality Magazine, May 2006 and May 2007.

Invited

- Doiron, T. "The history and future of gauge blocks", METROMETE, Bilbao Spain, 2008.

Customers:

- The identities of our measurement service customers are protected by our privacy policy. In the period of FY-05 to FY-09 the DMP provided dimensional measurement services to 360 different industrial customers and 25 government entities. The DMP calibrates over 5,000 master gauges, instruments, and artifacts per year with gross revenues of approximately $0.8M per year.

Collaborators:

- In selected situations, the program makes available sanitized calibration results to U.S. manufacturers of artifacts that have been recently calibrated in order to improve their quality assurance process and competitiveness.

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Improvements in Calibrations and Uncertainty Evaluation

Industrial Need:

Practical access to the SI unit of length for US manufactures requires continuous upgrades to the DMP dimensional calibrations to maintain state of the art measurement capabilities needed for international competitiveness. Understanding the evaluation of measurement uncertainty is crucial to improved measurement accuracy for accredited laboratory measurement.

Project Objective:

Provide industry with higher accuracy dimensional calibrations and more technologically relevant calibrations to enhance U.S. productivity and quality. Effective measurement services require that ongoing research into improvements to high accuracy calibrations be continually done. This research focuses on reducing the uncertainty in DMP measurement results or the creation of new DMP calibration services that support high value or high leverage calibrations for the US industrial customer base. Additionally, the project will develop new methods to quantify and standardize the evaluation of measurement uncertainty which is crucial to improved measurement accuracy.

Technical Approach:

Improving dimensional calibrations through reduced uncertainty, greater throughput, and new measurands is a core mission of the DMP. This project develops these new capabilities through acquiring new equipment, characterizing measurement processes, and developing documentation and quality assurance activities for improved measurement services. Specific calibration improvements include upgrading contact surface roughness capability through new instrumentation, improving tape measurement accuracy, characterizing gauge penetration effects, and increasing throughput of flatness measurements. Activities in measurement uncertainty characterization include: advancing the state of the art in uncertainty evaluation theory, providing documents on the practical access to measurement traceability, and developing new evaluation procedures compliant with the international Guide to the Expression of Uncertainty in Measurement (GUM). The project also advances the standardization of measurement uncertainty evaluation, for both DMP calibrations and industrial practice. DMP staff are active members of national (ASME B89.7) and international (ISO TC213 WG4 and JCGM WG1) standard committees and have extensively lead standardized developments in this area. Additionally, basic research into measurement uncertainty, in cooperation with NIST's ITL, addresses fundamental issues in this field.

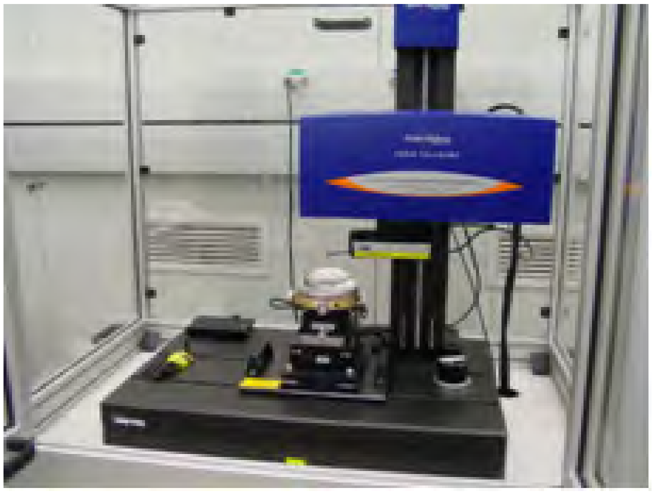
New Equipment and Measurement Capabilities:

High accuracy long length measurement capability to calibrate 1 km of fiber optic cable with an uncertainty of less than 2 parts in 10^{-6}.

UMAP multi-sensor coordinate measureing machine (CMM) which extends range of small feature calibration below 50 µm.

Nikon NEXIV 6555 multisensor (vision, triangulation) system. System is used for miscellaneous measurements such as the standard scales used in forensic photography.

Mahr ULM long waybed micrometer installed in AML for use in routine measurements of sphere, cylinder and ring calibrations.



AMETEK / Taylor-Hobson Form Talysurf PGI 1240 to be used in surface finish calibrations.

Moore Special Tool Model 1440 indexing table that will be used to simplify angle calibrations and make the calibrations more efficient.

The second Moore M48 CMM was installed in the AML and is currently being error mapped. It should be on line for measurements by winter, 2009.

The tape measurement system has now been outfitted with a new measurement carriage have electronic levels and a wireless modem to provide automatic pitch correction. The facility is also undergoing a significant thermal control upgrade. The measuring tape facility has reduced its uncertainty resulting in more accurate master tapes calibrated at NIST: providing metrological traceability to the SI unit of length for the millions of tapes manufactured in the US each year.

Impact/Benefits

Over the last 5 years the calibration facilities have had major changes which included better environmental control laboratories, new equipment which has extended the scope of our capabilities into new critical areas of the US measurement system. The increased demand for calibrations and increased income show this trend. There has also been a rising number of Special Tests, high accuracy one-of-a-kind measurements. PED staff have spent considerable effort giving presentations at industry meetings showing our new capabilities.

Measurement uncertainties for rings, plugs, and tapes have decreased. New measurement capability for measuring long cables by managing frictional effects have allowed 1 km optical fiber length standards to be calibrated at unprecedented accuracies. An entire series (ASME B89.7)

......advances the standardization of measurement uncertainty evaluation, for both DMP calibrations and industrial practice.

of US standards on measurement uncertainty has been published. The US has been represented at both ISO and BIPM/JCGM committees and has several standards in draft form. Research results on the finite resolution of measurement results have resolved the ISO 14253-2 debate.

Accomplishments:

- Changed paradigm for thermal control of dimensional gages from thermal isolation to convective heat transfer. Reduced times for "soaking in" and reduced thermal gradients between master and test gages by more than half.
- Second Moore M48 CMM installed and currently being mapped.
- Developed calibration techniques for new roundness instrument, lowering the uncertainty below 4 nm.
- Purchased Mitutoyo UMAP multisensor CMM with both vision and small feature (<100 nm) range.
- Installed new Nikon Nexiv 6555 multiprobe CMM with vision and point triangulation non-contact probes.
- Installed new Mahr long waybed 1D measuring machine.
- Provide calibrations with new contact stylus instrument for roughness and step height with the nano-scale surface profiler.
- New tape bench system restarts tape calibrations at higher accuracy due to improved thermal control.
- Complete installation of the wireless tape carriage with automatic pitch compensation for tape calibrations; improve bench accuracy.
- Submission to ASME (as project team chair) of a final draft copy of new revision of ASME B46.1-2009, Surface Texture (Surface Roughness, Waviness, and Lay)
- Completion (NIST is primary author and taskforce chair) US national standard: ASME B89.7.3.2 -2007, Guidelines for the Evaluation of Dimensional Measurement Uncertainty — provides a simplified approach to the evaluation of dimensional measurement uncertainty
- Completion (NIST is primary author and project team chair) ASME B89.7.4.1-2005, Measurement Uncertainty And Conformance Testing: Risk Analysis — provides guidance on the risks involved in any product acceptance / rejection decision.
- Completion (NIST is primary author and project team chair) ASME B89.7.5-2006, Metrological Traceability of Dimensional Measurements to the SI Unit of Length — describes the issues associated with claiming measurement traceability and provides a checklist for asserting metrological traceability of dimensional measurements
- Completion (NIST is primary author and project team chair) ASME B89.1.7-2009, Performance Standard for Steel Measuring Tapes
- Replace Pulfric Interferometric viewer with new phase shifting interferometer.
- The LSCMG oversaw the installation and commission of a new air handler in the NIST Tape tunnel laboratory facility. The increased air flow reduces the static spatial gradient from ±0.3 °C to approximately ±0.17 °C.
- Tyler Estler is principal author of JCGM 106, a high-level guide to the role of measurement uncertainty in conformity assessment. This work is carried out at BIPM in the Joint Committee for Guides in Metrology (JCGM), chaired by the BIPM Director.

Publications:

2009

- Stone, J. A., Decker, J. , Gill, P. , Lewis, A. , Juncar, P. , Rovera, D. , Villiseid, M., "Advice from the CCL on the use of unstabilized lasers as standards of wavelength: the helium-neon laser at 633 nm", Metrologia, Volume 46, pp. 11-18, 2009.
- Stoup, J., "Measurement of Large Silicon Spheres Using a Coordinate Measuring Machine" for a chapter in the new edition of the book "Coordinate Measuring Machines and Systems" being revised by Robert

Hocken, and to be published late 2009.

- Phillips, S.D., Estler, W.T. and Baldwin, J., July 2009. Economics of Measurement Uncertainty and Tolerances, Proceedings of the ASPE 2009 summer topical meeting, Peoria IL

2008

- Phillips, S.D., Toman, B., and Estler, W.T., "Uncertainty Due to Finite Resolution Measurements", Journal of Research of the National Institute of Standards and Technology, 113, 143-156, 2008.

2006

- Machkour-Deshayes, N. , Stoup, J. R., Lu, J. , Soons, J. A., Griesmann, U. , Polvani, R. S., "Form-Profiling of Optics Using the Geometry Measuring Machine and NIST M-48 CMM", NIST Journal of Research, 2006.

Presentations:

- Blackburn, C., "Toward a Standardized Approach for the Evaluation of Hand-Held Laser Scanner Performance", Association of Coordinate Metrology Canada, Windsor, Ontario, June 26, 2008
- Doiron, T, McLaughlin D., Schneider, A., "Use of Air Showers to Reduce Soaking Time for High Precision Dimensional Measurements", NCSLI Workshop and Symposium, St. Cloud MN, 2007
- Doiron, T., "Tutorial on Gage Block Metrology", 3 hour session at the Manufacturing Metrology Conference and Workshop in Clearwater, FL, 2007.
- Doiron, T., Half day uncertainty class at regional NCSLI meeting in Syracuse, NY, 2007.
- Doiron, T. "Torque and dimensional measurements", special NIST organized meeting for industry manufacturers and users of torque measurement systems, 2007.
- Dagalakis, N. G., Kim, Y.S. , Sawyer, D. S., Shakarji, C. M., "Development of Tools for Measuring the Performance of Computer Assisted Orthopaedic Hip Surgery Systems", Proceedings: Proceedings of the Performance Metrics for Intelligent Systems, Gaithersburg, MD, August 29-30, 2007.
- Londono, C., Lawall, J. R., Postek, M. T., Stone, J. A., Stoup, J. R., "The NIST Advanced Measurement Laboratory: at the Leading Edge of Measurement Science and Technology", Interferometry XIII: Techniques and Analysis, Proceedings: Proceedings of SPIE, San Diego, CA, August 14, 2006.
- Ma, L., Low, S. R., Song, J. J., "Investigation of Brinell Indentation Diameter From Confocal Microscope Measurement and FEA Modeling", Proceedings: Proceedings of Hardiness Measurements Theory and Application in Laboratories and Industries| 2007 |HARDMEKO, Tsukuba, Japan, November 19-21, 2007.
- Shakarji C. M., "Special Issues in Testing Uncertainty Evaluating Software." NMIJ (Japan). 2006
- Shakarji C. M., "Calibration of a Computer Assisted Orthopedic Hip Surgery Artifact", Proceedings Performance Metrics Conference, NIST Gaithersburg, October 2008
- Song, J., Vorburger, T. V., "Verifying Measurement Uncertainty Using a Control Chart With Dynamic Control Limits", Proceedings: Proceedings of 2007 Measurement Science Conference, Long Beach, CA, January 22-26, 2007.
- Song, J., Vorburger, T. V., "A Novel Parameter Proposed for 2D and 3D Topography Measurements and Comparisons", Advanced Characterization Techniques for Optics, Semiconductors, and Nanotechnologies III , Proceedings of SPIE Volume: 6672, San Diego, CA, August 26-30, 2007
- Song, J. , Low, S. R., Ma, L., "Tolerancing Form Deviations for NIST Standard Reference Material (SRM) 2809 Rockwell Diamond Indenters", Recent Advancement of Theory and Practice in Hardness Measurement | 2007 | | HARDMEKO 2007, Tsukuba, Japan, November 19-21, 2007.
- Song, J., Vorburger, T. V., Ballou, S. M., Renegar, T. B., Ma, L. , Whitenton, E. P., Kelley, D. R., Clary, R. A., Zheng, A. , Ols, M. , "NIST Standard Bullets and Casings Project "Proceedings of 2007 ASPE, Dallas, TX , October 14-19, 2007.
- Song, J. , Vorburger, T. V., "Topography Measurements and Applications", Proceedings of the 2006 International Symposium on Precision Mechanical Measurements (ISPMM), Urumqi, China, August 03-06, 2006.
- Stone, J. A., "Using uncalibrated lasers as wavelength standards", Proceedings, Simposio de Metrologia, Queretaro, October 22-24, 2008.
- Stone, J. A., Lu, L., Egan, P., "Calibrating Laser Vacuum Wavelength With a GPS-Based Optical Frequency Comb", Proceedings: Proceedings of NCSL Workshop and Symposium 2007, St. Paul MN, May 15, 2007.
- Stoup, J., "A Few Case Studies in Uncertainty Using the NIST M48 CMM" published in the proceedings

of the ASPE Spring Topical Meeting, Mechanical Measurements and Measurement Uncertainty, Albuquerque, NM, United States, 2009.

- Stoup, J., " High Accuracy CMM Measurements at NIST", Mexican National CMM Users Meeting at the Volkswagen Production facility in Puebla, Mexico, 2007.
- Stoup, J. "Stretching and Shrinking the Measuring Volume of the NIST M48 Coordinate Measuring Machine", Measurement Science Conference in Anaheim, CA, March 14, 2008.
- Stoup, J., "Early Results from the NIST M48 CMM in the New AML Facility", Measurement Science Conference in Anaheim, CA, March 2, 2006.
- Stoup, J., "An Enhanced Thermal Environment for the NIST M48 Coordinate Measuring Machine," 2005 NCLSI Conference and Symposium, Washington DC, August 2005.
- W. T. Estler, Tutorial: "Principles and Applications of Measurement Uncertainty", ASPE Annual Meeting, Dallas, Oct 2007
- W. T. Estler, "ASME B89 Standards on Measurement Uncertainty and Traceability", ACMC Conference, Rochester, May 2007
- W. T. Estler, "US Dimensional Metrology Standards Activities", CIRP Committee STC P, Manchester, England, Aug 2008
- W. T. Estler, Tutorial: "Self-calibration: Reversal, Redundancy and Absolute Testing", ASPE Annual Meeting, Portland, Oct 2008
- W. T. Estler, "Uncertainty Concepts and High-accuracy Dimensional Metrology",
- Remmele/Lockheed engineering group, Feb 2005
- W. T. Estler, "Uncertainty Concepts and Decision Making", Joint B89/Y14.5 meeting, Minneapolis, May 2005
- W. T. Estler, "US Dimensional Metrology Standards Activities", CIRP Committee STC P, Antalya, Turkey, Aug 2005
- W. T. Estler, Tutorial: "Self-calibration: Reversal, Redundancy and Absolute Testing", ASPE Annual Meeting, Norfolk, Oct 2005
- W. T. Estler, "The Joint Committee for Guides in Metrology: Supplementing the GUM", ASPE Summer Topical Meeting, State College, PA June 2004
- W. T. Estler, Tutorial: "Principles and Applications of Measurement Uncertainty", ASPE Annual Meeting, Orlando, Oct 2004

SRM/RMs produced

- Calibration of a number of optical fiber diameter SRM 2553 and 2554.

Awards

- S.D. Phillips and W.T. Estler, 2009. ASME's Certificate of Achievement" for outstanding standards work
- S. D. Phillips, W. T. Estler, B. R. Borchardt, C. Blackburn, D.S. Sawyer, The Judson C. French Award, For significant improvements in accuracy and extensions of range in the calibration of long length standards

Customers

- The primary customer of this project is the DMP calibration program which will offer these new capabilities to our industrial calibration customers. Additionally, the general metrology community benefits from improvements in theoretical infrastructure provided by extensions and improvements in the GUM and related uncertainty standards.

Collaborators

- Michigan Metrology, Detroit, MI
- Taylor Hobson, Chicago, IL
- Veeco Metrology, Tucson, AZ
- Zygo Corporation, Middlefield, CT
- Son Bui, Ph.D., Tucson, AZ
- PTB, Germany
- BIPM, JCGM WG1
- ASME B89.7 committee, TC213 WG4 and WG10

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Optical Comb and Refractometry

Industrial Need:

Industrial measurements steadily require increasingly accurate measurements from NIST. This accuracy originates with the realization of the SI meter and this project will allow the DMP to more accurately and compressively realize the meter. Additionally, laser interferometry as practiced in industry has two needs addressed by this project. First, there is a need for better determination of the refractive index of air, because the refractive index determines the laser wavelength (in air) and is the basic limitation on the accuracy of interferometric length measurement. Second, there is a need to know the wavelength of lasers that do not operate at the usual red wavelength, so as to satisfy the needs of multicolor interferometry or to enable new, novel measurement systems. Use of the comb will lower the barrier to innovation by allowing common telecommunication lasers to be easily calibrated by receiving GPS satellites frequencies as the reference oscillator for the comb. Additionally, it could allow frequency dissemination via an all-optical fiber telecommunication network.

Project Objective:

This project will advance the realization of the meter through the use of absolute refractometry to achieve below one part in 10^8 accuracy. This will reduce a major uncertainty source in high precision length measurements. The meter is currently realized by using a small set of frequency calibration optical sources, and corrected for the index of refraction using measurements of temperature, pressure, and humidity via the Edlén equation. This technique is limited to several parts in 10^8. This effort will halve this uncertainty, furthermore, by using an optical comb, a near continuous set of calibrated frequency sources will be available from the infrared to the visible, allowing advances in multi-frequency interferometry needed to reduce the measurement ambiguity interval. Realization of practical frequency standards from the comb are expected by 2011, while industrial in situ availability will be available some years following.

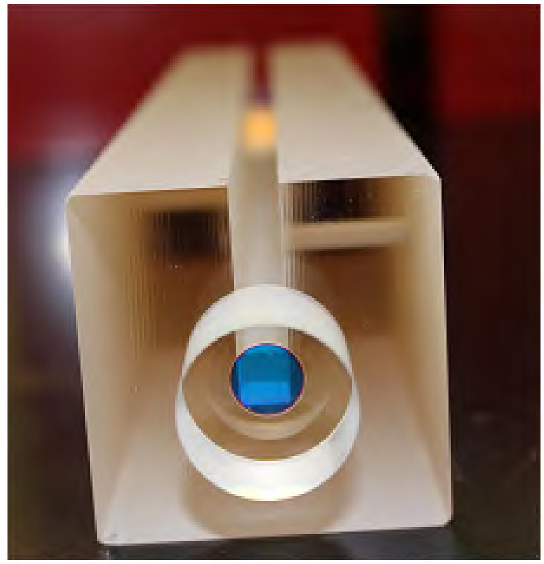
Technical Approach

The first goal of this project focuses on the practical realization of the SI meter in air. We will overcome the index of refraction barrier that limits wavelength corrections to around 50 parts in 10^9 through the use of an absolute refractometer. The refractometer consists of an extremely stable optical cavity that measure changes in the optical path length to a few nanometers. It is anticipated that deployment of the refractometer into other DMP calibration instruments, including the Moore M48 CMMs, will reduce the measurement uncertainty associated with the interferometry to a negligible contribution, in contrast to it being a major uncertainty source in the current systems.

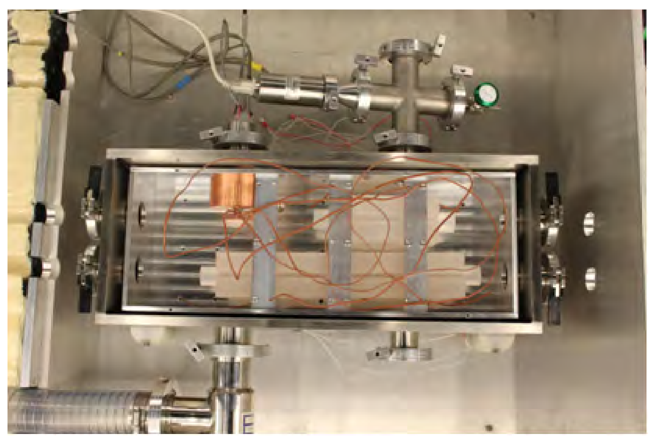
The second goal of this project is the practical realization of calibrated optical wavelengths using the optical comb. In this arrangement, the meter is realized by using the atomic clock aboard GPS satellites, accessed via their broadcast frequencies, and used as a reference frequency for the comb. This effort will demonstrate that an extremely high accuracy optical reference source can be created in a manner that can be realized in industry. Additionally, the comb has the potential to serve as a multiple wavelength source for multicolor interferometry.

Equipment Composing the Optical Comb

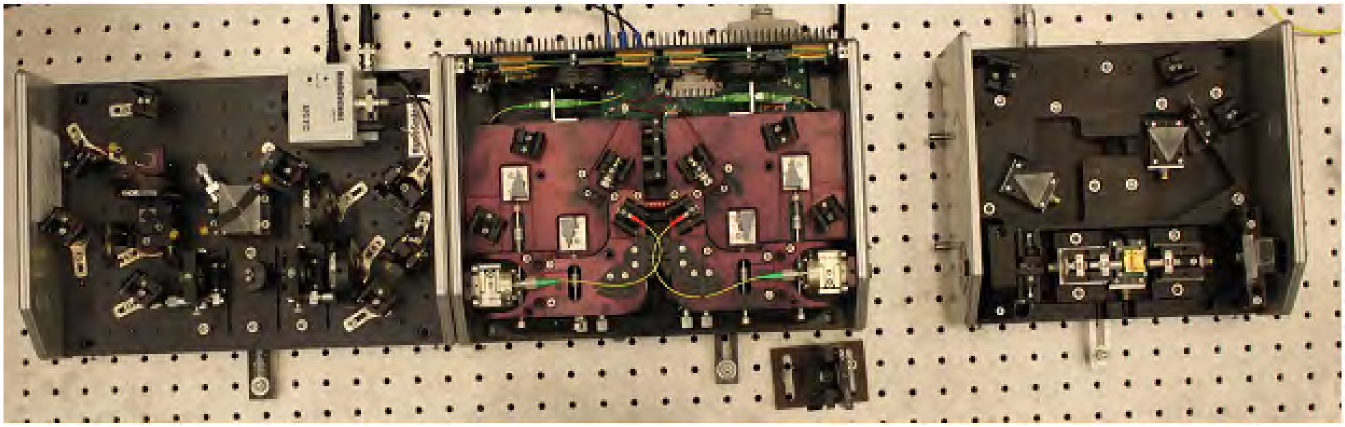
GPS antenna (mounted on exhaust pipe structure). GPS provides the basic frequency standard underlying comb measurements. It can deliver 1 part in 10^{12} uncertainty and is continuously "calibrated" (monitored) by NIST Boulder.



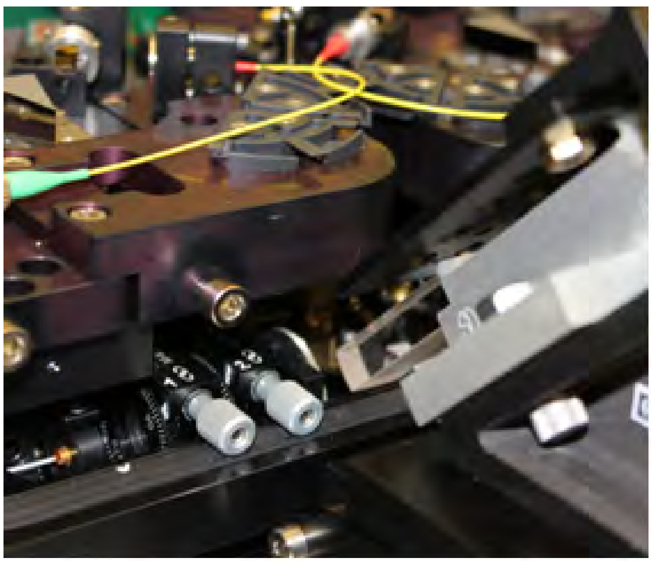
Optical Cavity for refractive index measurement. Two highly reflective mirrors are bonded to a long block of ULE (ultra-low-expansion) glass with a channel cut lengthwise in the glass. (The front mirror surface appears blue in the picture.) The channel region between the mirrors is completely open to the surrounding air, and the optical length between the mirrors depends on the refractive index of the air.



System for testing refractive index measurement. The square light-colored objects are two optical cavities suspended in an environmental/vacuum chamber where the temperature and pressure of a gas can be controlled. Several layers of thermal shielding reduce temperature gradients to negligible values, so that the two cavities can be compared to each other with very high accuracy, an important step in verification of the method. Visible in the picture are thermocouple wires going to a reference junction—the large copper block in the side of the chamber. The reference temperature is measured with a precision thermistor.



Optical frequency comb. The central section includes a femtosecond laser, amplifiers, and highly nonlinear fibers to spread the spectrum. On the left is the f-to-2f interferometer used to control the offset frequency. On the right is a frequency doubler, to convert infrared to visible light.



Tuning the comb repetition rate. Glass mounted on guiding mechanism slides in or out of free-space region of femtosecond laser, adding optical length to the resonator and changing the repetition frequency.

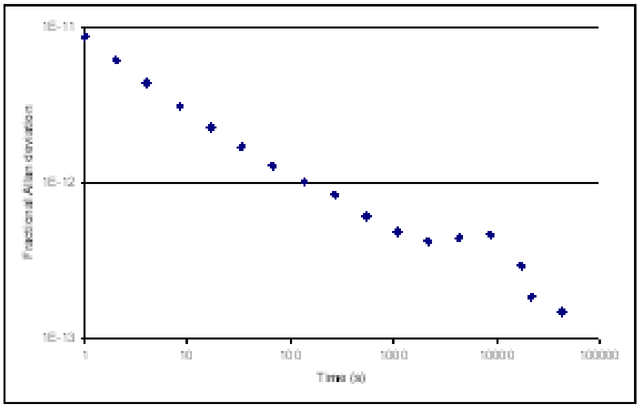
there is a need for better determination of the refractive index of air, because the refractive index determines the laser wavelength (in air) and is the basic limitation on the accuracy of interferometric length measurement.

Impact/Benefits:

- This project is still in the development phase however several publications describing the basic science discovered have been produced.

Accomplishments:

- Comb and GPS system installed in AML [2006]
- Comb: Allan variance (a stability measure analogous to relative standard deviation) falls below 1 part in 10^{12} when averaging over 150 s or longer. Accuracy is significantly better than iodine stabilized laser and will meet all anticipated needs. Best performance ever reported for a GPS-controlled system.
- Comb: Used for first internal calibration—to verify performance of our iodine stabilized laser.
- Refractive Index: Developed computer control system to automatically lock a laser to a cavity, and pattern recognition of a video image to automatically distinguish between axial and off-axis cavity modes.
- Comb: Methods developed for continuing verification of performance of GPSDO and various aspects of comb operation. Published paper describing how to use internal consistency checking to verify comb



Allan deviation (measure of relative stability) falls below 10^{-12} at 150 s. The fact that it continues to fall for longer times provides evidence that the GPSDO is working properly.

measurement uncertainty at the 10^{-12} level.

- Comb: Coverage of visible spectrum achieved with addition of two PPLN crystals, allowing second harmonic generation from the green (543 nm) to deep red. (Infrared operation prior to frequency doubling is also possible.)
- Comb: Developed single-arm f-to-2f interferometer as a more robust replacement for dual-arm mechanism. (Has provided trouble-free measurement/locking of frequency offset for more than a year.)
- Comb: Further confidence in the GPSDO attained via a careful set of comparisons to a second GPS system at NIST.
- Refractive Index: A fast control loop-- Pound-Drever-Hall (PDH) frequency stabilization-- demonstrated for laser locked to cavity, providing precise short-term tracking of refractive index changes.
- Refractive Index: A second (slower) thermal control loop demonstrated, working in conjunction with the PDH so that refractive index can be tracked over the 1.3 GHz tuning range of the laser.
- Refractive Index: Demonstrated a very high level of intensity stabilization of lasers, using acoustic-optic modulator for intensity control (0.005%) • Refractive index: Inner shield box to eliminate thermal gradients was designed and fabricated.
- Comb: Frequency measurement signal-to-noise improved via various strategies, including careful selection of wavelength range with a diffraction grating.
- Comb: Completed suite of internal testing capabilities with a new method for realtime detection of frequency miscounting.
- Comb: Developed new method for tuning the comb repetition frequency, easing the process of establishing the "order" of a comb component. We can now say that all elements are in place for comb calibrations.
- Comb: On the basis of previous bullets, we have fully characterized GPS-based comb with wavelength capability throughout the visible.
- Refractive Index: New optical cavities installed in environmental/vacuum chamber with less than 3 mK temperature gradients in inner chamber. Two Pound-Drever-Hall servos lock lasers to track refractive index
- Comb: Paper in progress to describe new NIST procedures that can be used by anyone with access to a GPS signal to make guaranteed (arguably traceable) laser frequency measurements, thus realizing 'Length by satellite".

Publications:

- Stone J., Lu L, and Egan P., 2007 "Calibrating Laser Vacuum Wavelength with a GPS-based Optical Frequency Comb" Measure 2, 28-38, (Dec. 2007).
- Stone J., Decker J., Gill J., Juncar P., Lewis A., Rovera G. D. , and Viliesid M, 2009 "Advice from the CCL on the use of unstabilized lasers as standards of wavelength: the helium-neon laser at 633 nm" Metrologia, 46 2009 11-18.
- Stone J. and Egan P. "A comb tied to GPS for NIST laser calibrations" to be published, (in progress)

Presentations:

- Stone J., Lu L, and Egan P., 2007 "Calibrating Laser Vacuum Wavelength with a GPS-based Optical Frequency Comb", Proc. of NCSL 2007 Workshop and Symposium, St. Paul, July 29 to Aug 2 2007.

Customers:

- The optical comb will serve internal needs as the top of the traceability chain in PED.
- There are both internal and external (DOD) needs for calibration at 543 nm, and the multicolor capabilities of the comb will fulfill this need.

- For refractive index, our most pressing needs are internal, where state-of-art refractive index measurements will be needed to meet future industrial needs, particular for the semiconductor industry where refractive index corrections limit our ability to deliver measurements of the highest accuracy.
- Measurement of refractive index also has potential applications to other types of NIST and industry measurements; for example, there is a clear potential to use refractive index for pressure measurements, of interest both to the NIST pressure group and to DOD.
- Boeing is already requesting that we provide traceability for their iodine stabilized laser used as a primary length standard.

Collaborators:

- NIST, Boulder

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MicroFeature Calibration Development

Industrial Need:

Micro-parts are becoming increasingly important to our economy, but systems for their measurement are still in their infancy. An example of an immediate need is for measurement of advanced fuel injectors (holes of ≈100 µm diameter and below) from manufacturers such as Caterpillar and Cummins. In past years NIST has also received requests for measurements of micro-optical switching components that we could not (at that time) satisfy, but we can now satisfy this need and hope to serve the needs of the telecom industry or other users of fiber-based micro-optical components. More generally, we expect to see a constant stream of emerging needs as a consequence of the strong continuing trends towards miniaturization and maturation of the MEMS industry

Project Objective:

Develop multiple approaches for high accuracy measurements of small features (1 mm to 0.1 mm). This research focuses on extending the range of our ultra-high accuracy coordinate measuring machines to feature sizes less than a millimeter with the goal of measurements as small as 10 µm, and uncertainties below 100 nm. A central aspect of this project is the deployment of a second Moore M48 high accuracy CMM dedicated to microfeature calibrations. DMP will install the new M48, develop metrological capabilities similar to the current M48, and then adapt the new M48 with the recently developed fiber microprobe and a vision system (as described below). DMP will have this new CMM characterized and approved by the PED quality system for both micro-probing and vision calibrations by Q1/2012. A secondary goal of the effort is the continuous measurement improvement of the current UMAP microfeature CMM, used in measuring features such as fuel cell micro-channels.

Technical Approach:

The DMP will develop and provide high accuracy metrology for an array of high value products in medical, aerospace, automotive, energy, and biotechnology fields that include small features ranging from 10 µm to 1000 µm in size. While two-dimensional metrology in this domain has long been available, three-dimensional coordinate metrology is not currently capable of the levels of accuracy required. Specific challenges include measuring the full geometry of 100 µm bores with aspect ratios of 20:1, as found in next generation fuel injectors. In addition, to access many of these small features, DMP will develop methods for sub-microNewton probing forces to avoid damage to the part. Additionally, the DMP will study the physical issues such as electrostatic forces, meniscus forces, and physisorbed films that represent phenomena that can bias high precision measurements. This effort will provide microfeature measurement services with 100 nm or less uncertainty by 2011.

Equipment:

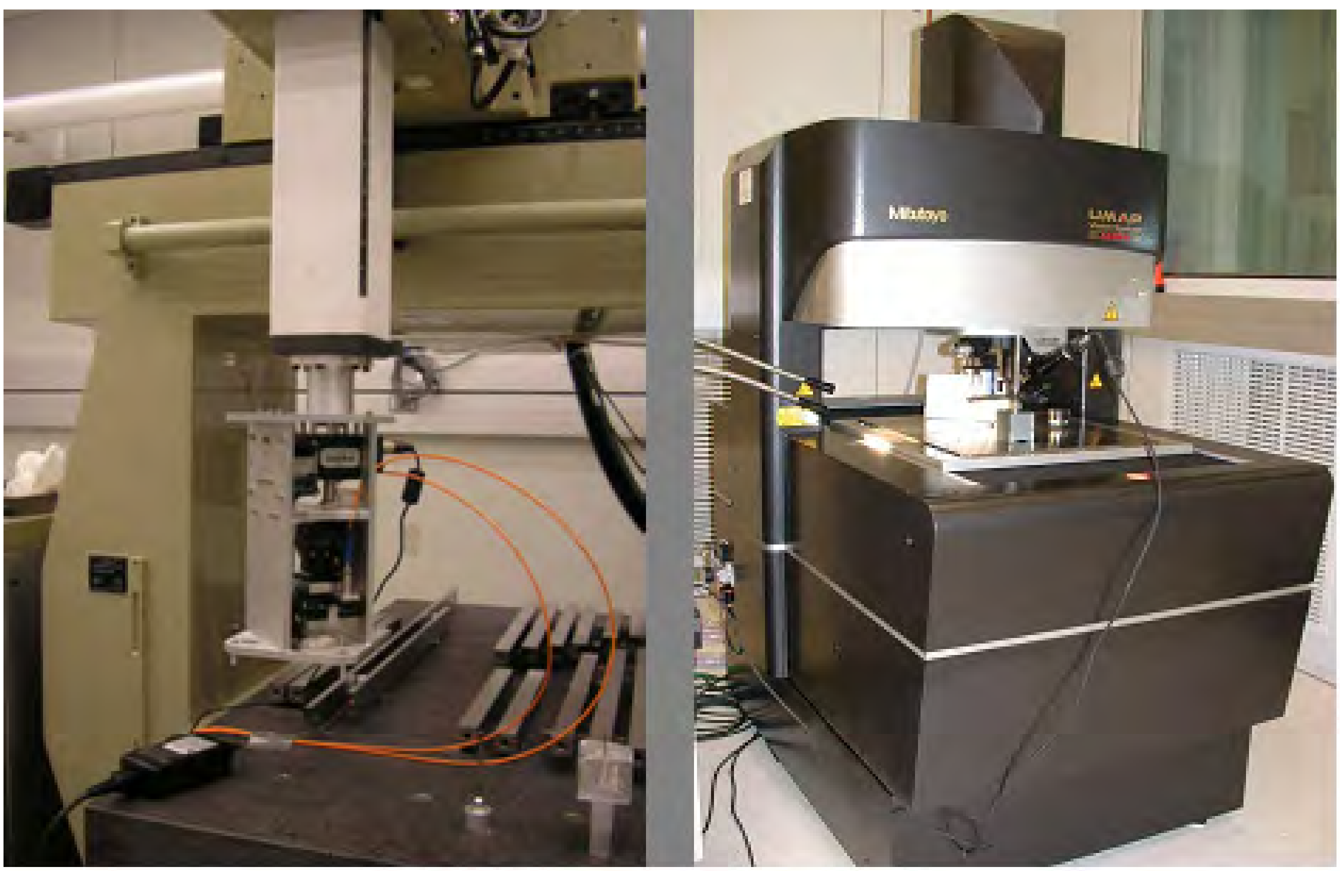
Most operations are currently centered around our M48 CMM (Moore Special Tool), with a second M48 in the process of being brought on line. In addition, some measurements—most notably fuel cell channels and nozzles—have been carried out on our Mitutoyo UMAP CMM, which can perform measurements with either a vibrating fiber probe or a vision system. Our CMMs are shown in figure 1, and some of our probing systems are shown in Figure 2.

Impact/Benefits:

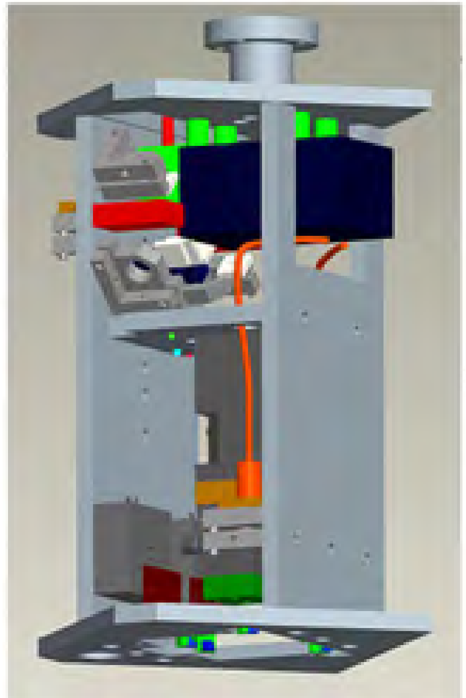
The new capabilities have opened up entirely new industries as customers. We have made high accuracy measurements on small nozzles and fuel injectors, center hole of fiber ferrules, very small volume standards, LIGA-produced gears, areas of knife edge apertures, and many other artifacts. Measurements of the holes in fuel injectors make it possible to carefully quantify the errors associated with current measurement techniques (X-ray tomography). Our measurements of knife-edge apertures are perhaps the most accurate in the world, providing a path toward improved radiometry standards. Measurements of micro-pivots help to elucidate how form relates to function. Some fiber-probe measurements are illustrated in the section below.

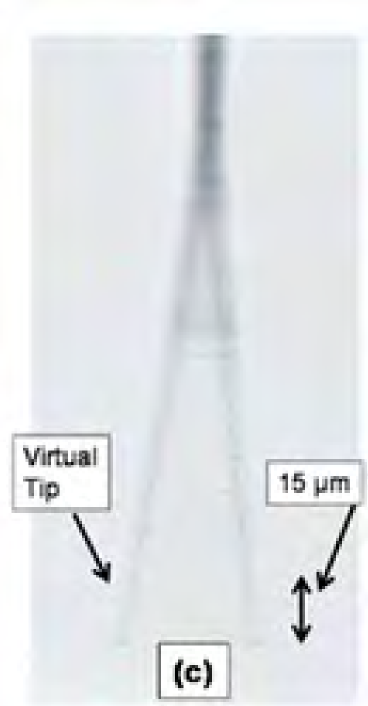
Accomplishments:

- Fiber probe: first measurements of 125 µm optical fiber ferrule
- Fiber probe: Cross-checks provide verification of performance to better than 70 nm; excellent agreement found between fiber probe and various other measuring



Coordinate Measuring Machines used for microfeature measurements. Left: The M48 (with NIST Fiber Probe). Right: Mitutoyo UMAP





Three probing systems. Left: Classic CMM probe (Movamatic) can measure sub-millimeter features. Center: Solid model of NIST fiber probe, which can measure holes smaller than 100 µm diameter. Right: vibrating stylus of InsituTec probe, with a potential capability to measure 20 µm holes. The UMAP also uses a vibrating probe, but of a different design.

techniques used at NIST, and between NIST and PTB measurements.

- Fiber probe: Developed a buckling technique for z-axis measurements with demonstrated 5 nm sensitivity; first measurements of a hemisphere demonstrate 3-d capability.
- UMAP: Characterized errors in system and began using this machine for practical measurement such as flow nozzles and fuel cells.
- Fiber probe: Vibration-assisted scanning speeds data acquisition and helps in collecting higher density form data.
- Fiber probe: measurement range extended to 10 mm; “hole reversal” tests developed to demonstrate that depth-related errors (stem-wall interactions, etc) are negligible.
- Fiber probe: Rebuilt with a new higher resolution camera and mounted on the ram of the machine.
- New M48: Procurement and delivery of new M48 to AML.
- Fiber Probe: Measurement of knife-edge apertures (radiometric standards) provide possibly best-in-world capability. Ultra-low probe force makes possible a measurement that was previously not possible. This is also first practical application of the spindle-based scanning system.
- InsituTec Probe delivered: began integration with M48 control system.
- Fiber Probe: Performed first NIST calibration using the NIST fiber probe to specific measurement tasks that have unusual requirements, including reverse-taper holes as in advanced fuel injectors.
- Fiber Probe: Developed new analysis tools for measurements of 3-D artifacts with non-circular probes.
- New M48: Fully operational with updated control software. As a test, calibration artifacts were measured successfully.
- New M48: Steel Moore table was removed and replaced with a 2ft x 4ft granite surface plate, increasing Z range of the machine and maximizing the measurement volume.
- New M48: Update of refractive index system completes task of making new M48 functional duplicate of the old M48 CMM.

- UMAP: Nearly a factor of 2 performance improvement follows upgrades of probe and environmental control.
- UMAP: System purchased by NIST (previously on loan).
- New M48: Completed error map of rotational error components. Straightness and squareness maps are currently in process.
- Fiber probe: Two papers and a conference proceedings review our small-feature research, document current state-of-art, discuss sources of error, and outline future directions for microfeature measurement.

"Thankfully, we found the NIST UMAP micro-probe capability. It perfectly fit our need because it was able to measure the small nozzle diameters that we have by physical touch and to a precision smaller than about 0.5 µm. For the first time, we now have a properly accurate calibration artifact for our optical system."

Daryl Roberts, MSP Corporation, Shoreview, MN

Publications List:

- Muralikrishnan B., Stone J., Stoup J. , and Sahay C. 2009 "Micro-feature dimensional and form measurements with the NIST fiber probe on a CMM", to be submitted to Cal Labs Magazine.
- Stone J, Muralikrishnan B, and Sahay C. 2009 "Geometric effects when measuring small holes with micro contact probes" to be submitted to NIST Journal of Research.
- Muralikrishnan B., Stone J., Stoup J. 2008 "Area measurement of knife-edge and cylindrical apertures using ultra-low force contact fiber probe on a CMM", Metrologia 45 281-289.
- Muralikrishnan B., Stone J., Stoup J. , 2006 "Fiber deflection probe for small hole metrology" Precision Engineering, 30 (2), 154-164
- Muralikrishnan B., Stone J., 2006 "Fiber Deflection Probe Uncertainty Analysis for Micro Holes", NCSLi Measure, September 2006. [Reprinted from proceedings of NCSLi]

Presentations

- Muralikrishnan B., Stone J., Stoup J. 2009 "Diameter and form measurement of a micro-hole in a fuel injector nozzle with the NIST fiber probe", Proc. Annual Meeting ASPE, Monterey (Sept 2009)
- Stone J. Muralikrishnan B. and Stoup J. 2008 "Microfeature measurements with a fiber probe: unique problems and their solution", unpublished, Mexico CMM users group at CENAM, Queretaro Mexico.
- Muralikrishnan B., Stone J., Stoup J. , 2007 "Roundness Measurements Using The NIST Fiber Probe" Proceedings of the Annual Meeting of the American Society for Precision Engineering
- Estler T. 2007 "Recent results using the NIST fiber probe" CIRP STC P, Dresden, Germany, 24 Aug. 24.
- Muralikrishnan B., Stone J., Stoup J. , 2006 "Enhanced Capabilities of the 'NIST Fiber Probe' for Micro Feature Metrology", Proc. ASPE.
- Stone J. Muralikrishnan B. and Stoup J, 2006 "A fiber-based probe for microfeature measurement", unpublished, 56th CIRP General Assembly, Kobe, Japan, August 20-26.
- Muralikrishnan B., Stone J., Stoup J. , 2006 "Microfeature Metrology", MicroManufacturing

Conference 2006, Society for Manufacturing Engineering, Los Angeles CA.

- Stoup, J. R., Faust, B. S., Doiron, T. D., "Early Results From the NIST M48 CMM in the New AML Facility", Proceedings: Measurement Science Conference, Anaheim, CA, March 02-03, 2006.
- Muralikrishnan B., Stone J., 2005 "Fiber Deflection Probe Uncertainty Analysis for Micro Holes", Proceedings of NCSLi Washington DC Aug 7-11, 2005
- Stone J, Muralikrishnan B, and Stoup J. , 2005 "Fiber probe for CMM measurements of small features", Proceedings of SPIE Vol. #5879 "Recent developments in dimensional measurements III" Editor(s): Jennifer E. Decker, Gwo-Sheng Peng, August 2005 p.254-264
- Muralikrishnan B., Stone J, and Stoup J. "Measuring Internal Geometry of Fiber Ferrules", , Proc. of the Micro Manufacturing Conference (SME), Minneapolis, MN, May 4-5 2005
- Muralikrishnan B. and Stone J., "Fiber Deflection Probe Uncertainty Analysis for Micro Holes" 2005 proceedings of NCSLi Washington DC Aug 7-11
- Awards: DOC Silver Award for fiber probe development, 2007

Customers:

- The most immediate need is for measurement of advanced fuel injectors from manufacturers of internal combustion engines.
- NIST has also received requests for measurements of micro-optical switching components which we could not perform previously but now have suitable capability; more generally, we hope to serve the needs of the telecom industry or other users of fiber-based micro-optical components.
- We expect to see a constant stream of emerging needs as a consequence of the strong continuing trends towards miniaturization and maturation of the MEMS industry; we performed our first measurement of a micro- gear this year and expect to see increasing demand for measurements of this type of artifact.

Collaborators:

- WT&T; Canada (fiber optics)
- InsituTec; Charlotte NC (CMM probes)
- MSP Corp Shoreview, MN (small holes)
- University of Hartford (probes)
- Optical Technology Division, NIST (knife-edge apertures)
- Mitutoyo of America (UMAP)
- DOE (fuel cells)
- Ceramics Division at NIST (Wolfgang Haller)

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Complex Geometry Instrumentation and Standards

Industrial Need:

Complex surfaces are increasingly employed in manufacturing, especially for large components. Not surprisingly a boom in instruments and methodologies to measure these structures is underway – which are rapidly replacing traditional measurement technology such as theodolites and large CMMs; the latter are immobile, represent a large fixed capital investment, and are slow at collecting the large number of data points needed to fully characterize these complex surfaces. Recent developments in electro-optical instrumentation eliminate these drawbacks and greatly increase throughput with lower labor costs.

A wide range of technologies such as multilateration, laser trackers, LADAR, and structured light are rapidly advancing due to the availability of high-speed electronics and inexpensive computer power. The US is a major supplier of frameless metrology systems used in large structures; however, demonstrating their metrological capability is problematic. In a recent workshop chaired by NPL, PTB, and NIST on large scale measurement systems, one of the summary findings stated "So far, no common procedures for the evaluation of measurement uncertainty or for performing an interim check are in existence for large scale measurement systems. In the near future, the rigorous implementation of quality systems, not just in the aircraft and automotive industries, but in a wide context will generate a huge need for action in this area."

Complex mechanical surfaces often act as the interface with their environments in dynamic structures such as airframes, turbine blades, and ship hulls. Small deviations in manufacturing or assembly prevent optimal function and cause inefficiencies that can consume large quantities of energy. Major manufacturers such as Boeing and Pratt & Whitney increasingly rely on measurements of complex surfaces by frameless measurement systems. Traditional methods such as large fixed CMMs represent too large a fixed capital investment and are not reconfigurable as required in a flexible manufacturing environment.

Due to the complexity, large measurement volume, and demanding environmental conditions required to evaluate large scale metrology instrumentation, there does not exist any commercial calibration facilities for high accuracy calibrations for this class of instruments. This impedes both the technological development of high technology companies that are developing these instrumentation and also industrial users that seek independent verification of their instruments capability. Additionally, the lack of national or international standards for this class of metrology instruments both represents a barrier to adoption of this technology by industrial users (market failure due to available information) and inefficient capital equipment expenses because users are unable to select the correct instrument for their needs.

Project Objective:

This project will promote innovation, reduction of time to market, and advance this technology by providing access to high accuracy calibrations and artifacts, research facilities, and documentary standards, applicable to large scale complex geometry instrumentation. Specifically, calibration facilities will be developed with world class measurement and calibration capabilities for laser trackers, laser scanners, and similar instruments. For cooperative target instruments (e.g. those using retroreflectors) the instrument's ranging calibration will have a traceable measurement uncertainty of less than one-half part-per-million. For noncooperative target instruments measurements, a ranging calibration capability of less than two parts-per-million uncertainty will be established. Additionally, volumetric measurement calibrations suitable for testing a wide class of instruments will be created and will be flexible enough to be rapidly reconfigured for a variety of measurement and research needs. Finally, based on research results of this project, a team of NIST experts will promote a long term effort to establish national and international standards for this class of instrumentation.

Technical Approach:

The principal goal of this project is to create a calibration facility that can accommodate a wide range of scanning instrumentation. The activity bifurcates into a calibration of the instrument's ranging technology (i.e. the instrument's metric of length) and a calibration of the full 3D measuring capability. The ranging calibration facility is under construction in the NIST tape tunnel and includes a range for cooperative targets (e.g. retroreflectors) that can be translated by a carriage over 60 meters of range. The carriage will be position will be accurately measured by a laser interferometer that is calibrated by the iodine stabilized laser. The interferometry will be corrected for index of refraction effects by a series of sensors calibrated by NIST primary measurement systems. Additionally, the evaluation and correction of carriage angular rotation will be performed by developing long range autocollomation technology. Noncooperative target locations will be established

".....no common procedures for the evaluation of measurement uncertainty or for performing an interim check are in existence for large scale measurement systems."

using fixed kinematic mounts that can be calibrated by interferomerty and fixed in a highly dimensionally stable manner. The project also supports – primarily through national and international documentary standards activity – conventional coordinate metrology such as coordinate measuring machines (CMMs) which are widely used throughout US industry.

The 3D geometry calibration facility will include both fixed 'monument" artifacts, and repositionable portable artifacts (e.g. large calibration bars). Standardized volumetric calibrations will be automated to reduce labor and hence cost. Artifacts that can be used by industrial metrologists – and satisfying standardized testing procedures – will be developed using the extensive experience of the large scale metrology group. Additionally, calibration instruments for the economical calibration of these multi-meter long artifacts will be produced based on the group's design of the 1D ball bar instrument.

Finally, the project will employ the group's well-positioned staff in national (ASME B89 and ASTM E57) and international (TC213 WG10) standards organizations to leverage its research finding into widely distributed documentary standards. This will be achieved through detailed modeling of the parametric errors in this class of instruments, analysis of testing positions that are sensitive to these errors and experimental conformation of the efficiency of the testing procedure.

Left; 1D noncooperative target test range composed of 100 mm diameter titanium balls with calibrated size, form, and locations. Right; members of the LSCM group and industrial customers testing the non-cooperative target 1D ranging facility

Impact/Benefits:

Thus far, the project has extensively analyzed laser tracker technology and the resulting research and development has resulted in the world's first laser tracker standard: ASME B89.4.19 "Performance Evaluation of Laser-Based Spherical Coordinate Measurement Systems." This standard is now used by all major laser tracker manufacturers and provides industrial users with a common set of specifications to both compare different tracker designs and to more efficiently employ capital equipment purchases. This standard is now being employed as the template for an ISO standard for laser trackers.

Additionally, the project has developed the new capability to calibrate – both for range and volumetric performance – laser tracker instruments to the ASME B89.4.19 standard. We have recently calibrated units for both users and manufacturers of the laser trackers. In particular, we have improved the product development capability that these technology companies can (and have) used to improve their instruments and shorten their product development time.

Finally, the project has led an ISO effort to develop a new international CMM standard which has resulting in an ISO standard that includes several detailed tests that the US industry requires. This has allowed the US, for the first time, to harmonize its national standard for CMMs with the ISO standard.

Accomplishments

National and International Standards

- Completed (as taskforce chair and primary author) International Standard on coordinate metrology ISO 10360.2 (2009) Acceptance test and reverification test for coordinate measuring machines (CMMs) — Part 2: CMMs used for measuring linear dimensions.
- Completed (as division chair and primary author) US National Standard on coordinate metrology harmonizing the US with the international community by the development and publication of ASME B89.4.10360-2 (2008) Acceptance test and reverification test for coordinate measuring machines (CMMs) — Part 2: CMMs used for measuring linear dimensions. This includes the entire ISO 10360.2 standard plus significant amounts of new material on thermal errors.
- Completed (as division and project team chair) ASME B89.4.19-2006, Performance Evaluation of Laser-Based Spherical Coordinate Measurement Systems — is the world's first standard for laser trackers and similar systems. The document contains all the information necessary to specify and test the performance of laser trackers plus tutorial information to understand the uncertainty sources of these instruments.
- Completed (as project team member) ASME B89.4.22-2004, Methods for Performance Evaluation of Articulated Arm Coordinate Measuring Machines — addresses specifications and testing of this class of multi-joint articulated CMM instruments

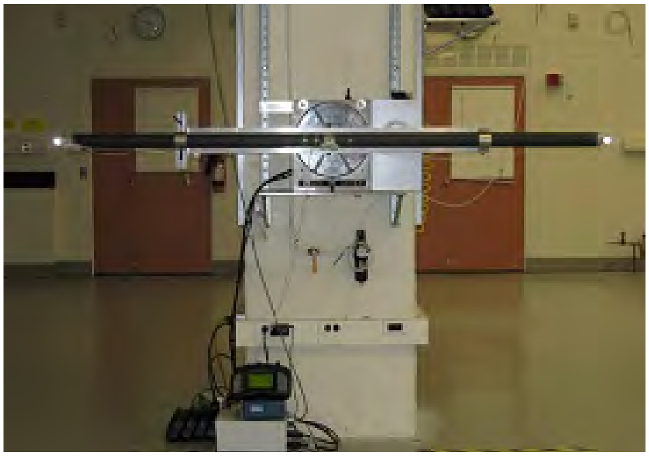
Left Commercial laser tracker under calibration in the large scale coordinate metrology lab with laser rail performing a volumetric performance test per ASME B89.4.19. Right: laser tracker under calibration for 1D cooperative target ranging test per ASME B89.4.19.

Calibration Facilities and Activities

- 2009 – initiated intercomparison of laser scanning test artifacts for ASTM E57.
- 2009 – First official NIST laser tracker calibration per the ASME B89.4.19 standard.
- 2009 – First official NIST articulated arm CMM calibration per ASME B89.4.22
- 2009 – Complete parametric error models of instruments using spherical coordinate systems developed.
- 2008 – Non-cooperative target 1D ranging facility begins initial testing.
- 2008 – Large scale flexible volumetric lab operational for 3D instrument characterization.
- 2007 – 60 m cooperative target 1D ranging facility operational.

Publications:

- Peggs, G.N., Maropoulos, P.G., Hughes, E.B., Forbes, A.B., Robson, S., Zeibart, M. and Muralikrishnan, B., 2009. Recent developments in large scale metrology, Proceedings of the Institution of Mechanical Engineers, Part B: Journal of Engineering Manufacture, 223 (6) p. 571-595.
- Phillips, S.D., Krystek, M., Shakarji, C., and Summerhays, K., 2009. Three-Dimensional Imaging Metrology: Dimensional measurement traceability of 3D imaging data, Proceedings of the SPIE, Volume 7239, pp. 72390E-72390E-7.
- Muralikrishnan, B., Sawyer, D. S., Blackburn, C. J., Phillips, S. D., Borchardt, B. R., Estler, W.T., 2009. ASME B89.4.19 Performance Evaluation Tests and Geometric Misalignments in Laser Trackers, Journal of Research of the National Institute of Standards and Technology 114, pp. 21-35.
- Muralikrishnan, B., Sawyer, D.S., Blackburn, C.J., Phillips, S.D., Borchardt, B.R., Estler, W.T. 2008. Performance Evaluation of Laser Trackers, Performance Metrics for Intelligent Systems Workshop, PerMIS'08, Gaithersburg, Maryland, August 19-21 (This article has been selected to appear as a Springer book chapter).
- Shakarji C. M., Morse E., Rhorer D., Phillips S. D., Slotwinski J., "2008: USCAR Digital-Virtual-Tools Team, Modeling of Compliant Parts Project", Phase 2 Final Report
- Muralikrishnan, B., Blackburn, C.J., Sawyer, D.S., Borchardt, B.R., Estler, W.T., Phillips, S.D., 2007. Laser Tracker Testing at NIST using the ASME B89.4.19 Standard, Journal of the Coordinate Metrology Systems Conference 2 (2), pp. 11-17.
- Dagalakis, N. G., Kim, Y.S. , Sawyer, D. S., Shakarji, C. M., 2007. Development of Tools for Measuring the Performance of Computer Assisted Orthopaedic Hip Surgery Systems, Proceedings of the Performance Metrics for Intelligent Systems (PerMIS)
- Dagalakis, N. G., Kim, Y.S., Sawyer, D. S., Shakarji, C. M. 2007. Design and Fabrication of an Operating Room Computer Assisted Orthopaedic Hip Surgery Artifact, Proceedings of the 2007 ASPE Annual Meeting.



Top: automated artifact for volumetric testing of laser trackers; lower: large scale volumetric lab

- Dagalakis, N. G., Stiehl, J. B., Kim, Y.S. , Sawyer, D. S., Shakarji, C. M. 2007. NIST Medical Phantom Device to Assist with the Calibration and Performance Testing of CAOS Systems, BONEZone, volume 6, number 3 page 18-29.
- Estler, W. T., Sawyer, D. S., Borchardt, B. R., Phillips, S. D. 2006. Large-Scale Metrology Instrument Performance Evaluations at NIST, The Journal of the CMSC, volume 1, issue 2, pages 27-32.
- Sawyer, D. S., Phillips, S. D., Borchardt, B. R., Estler, W. T. 2006. Recent Developments in the Standardization and Testing of Laser Trackers, Proceedings of the 2006 Manufacturing and Measuring Conference and Workshop.
- Phillips, S. D, 2005. Measurement Uncertainty, CMMs, and Standards: Today and the Future, Proceedings of 2005 International Dimensional Workshop Nashville, TN May 09-13.
- Weckenmann, A., Estler, W.T., Peggs, G., and McMurtry, D., 2004. Probing systems in Dimensional Metrology, Keynote Paper, Annals of the CIRP Vol. 53/2, 657-84.

Presentations:

Keynote

- Phillips, S.D. “Length Metrology: Cubits, Yards, and Meters”, Manufacturing & Measurement Conference and Workshop, May 2009, Orlando FL.
- Sawyer, D. S., “An Introduction to B89.4.19 Performance Evaluation of Laser Based Spherical Coordinate Measurement Systems”, Keynote address and Workshop, Large Volume Metrology Conference, Broughton, UK, November 1, 2005

Invited Talks

- Phillips, S.D, “Laser Trackers: Testing and Standards”, North American Coordinate Metrology Association, September 2009 Queretaro Mexico
- Phillips, S.D. Dimensional measurement traceability of 3D imaging data, SPIE conference on 3D Imaging Metrology (January 09)
- Phillips, S.D. “New Development in US and ISO CMM Standards” at the Coordinate Metrology Association Canada, June 2008 in Windsor Canada.
- Phillips, S.D. “Length Metrology: A Historical Perspective” ACMC July 2007 Rochester, New York.
- Phillips, S. D, Measurement Uncertainty, CMMs, and Standards: Today and the Future, Proceedings of 2005 International Dimensional Workshop Nashville, TN May 09-13, 2005

Other Talks

- Blackburn, C., “Toward a Standardized Approach for the Evaluation of Hand-Held Laser Scanner Performance”, Association of Coordinate Metrology Canada, Windsor, Ontario, June 26, 2008
- Sawyer, D. S., 2008 “NIST Progress in the Development of a Deployable High-Accuracy Artifact for Laser Tracker Performance Evaluation per ASME B89.4.19”, Coordinate Metrology Systems Conference, Charlotte-Concord, NC, July 21-25, 2008
- Phillips, S.D, and Estler, W.T, Principles and Applications of Measurement Uncertainty, Half day tutorial at the ASPE Annual Meeting, October 2007 Dallas Texas
- Phillips, S.D, “Laser Tracker Testing at NIST in Accordance with the ASME B89.4.19 Standard”, Coordinate Metrology Systems Conference, July 17th 2007 Reno NV
- Phillips, S.D, “New Developments in Laser Trackers:

Testing and Standards" at the Association for Coordinate Metrology Canada on June 21st 2007 Rochester New York.

- Phillips, S.D, "Laser Trackers: Testing and Standards" at the Manufacturing & Measurement Conference and Workshop on April 24th 2007 Clearwater Florida.
- Phillips, S.D, The Laser Tracker Standard and the NIST 60 m Ranging Facility, 3rd Annual NIST Workshop on the Performance Evaluation of 3D Imaging Systems (March 2-3, 2006) – a joint MEL – BFRL project
- Phillips, S.D, "Recent Developments in the Standardization and Testing of Laser Trackers", International Dimensional Metrology Workshop (IDW) 2006.
- Phillips, S.D, Measurement Uncertainty, CMMs and Standards; Today and the Future, Association for Coordinate Metrology Canada: June 9, 2005
- Sawyer, D.S. and Phillips, S.D, Laser Trackers, Standards, and Measurement Accuracy, Half-day tutorial at Coordinate Metrology Systems Conference (CMSC), Austin, TX July 19, 2005
- Phillips, S.D, Estler, W.T, and Ziegert, J., Principles and Applications of Measurement Uncertainty, Half day tutorial at the ASPE Annual Meeting, October 2004.
- W. T. Estler, "Report and Summary of the NPL Large-scale Metrology Workshop", CMSC, San Jose, July 2004
- W. T. Estler, "US Dimensional Metrology Standards Activities", CIRP Committee STC P, Krakow, Poland Aug 2004
- W. T. Estler, "NIST 1-D Range Test Facility", SPAR Conference, Houston, May 2006
- W. T. Estler, "Large-scale Metrology Instrument Performance Evaluations at NIST," CMSC Orlando, July 2006
- W. T. Estler, "US Dimensional Metrology Standards Activities", CIRP Committee STC P, Kobe, Japan, Aug 2006
- W. T. Estler, "Fundamentals of Traceability and Uncertainty", PTB Large-scale Metrology Workshop, Braunschweig, Germany, Nov 2006

Awards

- S.D. Phillips and W.T. Estler, 2009. ASME's Certificate of Achievement for outstanding standards work in coordinate metrology

Customers

- The target customers of this project are instrumentation companies that are developing new scanning technology and need access to calibration facilities to determine their actual measurement errors and thus enable improvements in the technology.
- A secondary customer base is the high end users of this technology who seek independent calibration documentation of their instruments. This group would include a wide array of industries including aerospace, heavy equipment, construction, and precision manufacturing.
- The broadest customer base is the general metrology community that will benefit from documentary standards to improve their capital equipment decisions.

Collaborators

- The Boeing Company; Seattle, WA
- Automated Precision Inc, Gaithersburg MD
- FARO Technologies, Kennett Square PA
- QuantaPoint Inc, Pittsburgh, PA
- Metris Inc., Manassas, VA
- BFRL, Materials and Construction Research Division

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Micrometer-Level Surface Finish Metrology

Industrial Need:

To develop improved speed, resolution, and accuracy of surface finish measurements to enable improved productivity in U.S. manufacturing. Secondly, to create a traceability system for optical inspection devices of bullets and casings in crime laboratories according to recently developed guidelines of the American Society of Crime Laboratory Directors (ASCLD).

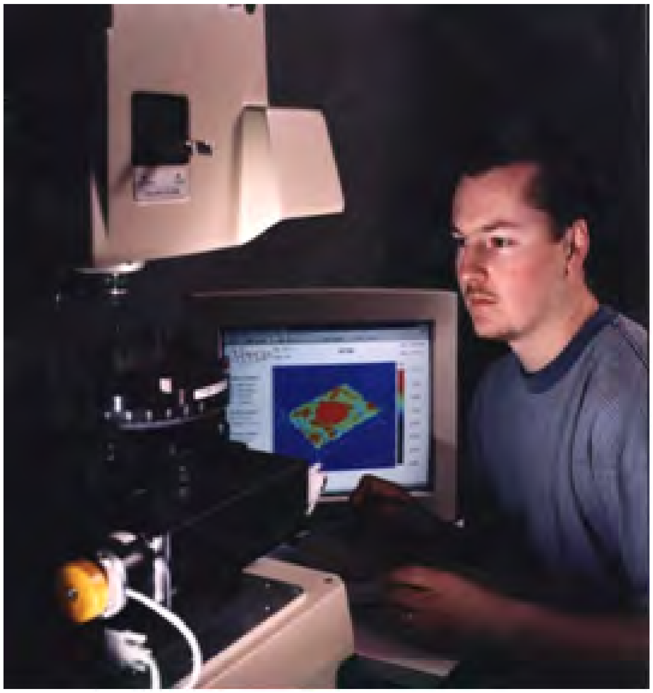
Project Objective:

Provide optical three-dimensional surface topography measurement at the micrometer level. Conventional contact stylus instruments are considered the "gold standard" for measurement of surface topography. However, advances in optical instrumentation enable rapid measurement of three dimensional (3D) surface topography. Characterization of these instruments and establishing their traceability at the micrometer scale is imperative to many industries and government agencies. Applications include the traceable measurement of hardness, where the shape of the indenter strongly affects results and the measurement of topography of bullets and casings in crime labs, where the fine individual characteristics that produce positive identification of individual weapons need to be separated from longer scale characteristics of overall shape.

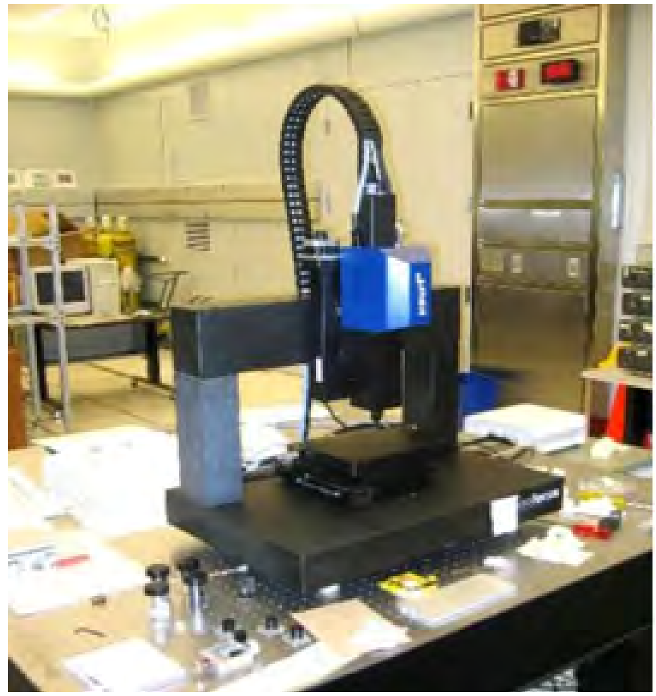
Technical Approach:

This project will establish the limits of validity for surface texture measurement obtainable by optical methods and will develop standards for measurement of surface texture and topography derived from optical measurements. The research directions include: 1) the measurement of surfaces using optical techniques and the comparison of the results with stylus methods, 2) leadership in the development of documentary standards for optical techniques, and 3) the development of physical standards and measurement parameters for optical microscopes used in crime labs to examine bullets and casings.

Specifically, the project will investigate the large errors arising with the use of coherence scanning interferometry for measurement of roughness average in the 50 nm to 300 nm range. This technique will be compared with that of confocal microscopy and the more fundamental stylus techniques. Additionally, documentary standards for phase shifting interferometric microscopy,



Veeco NT2000 Interferometric Microscope for measurement of surface texture by phase shifting interferometry and coherence scanning interferometry.

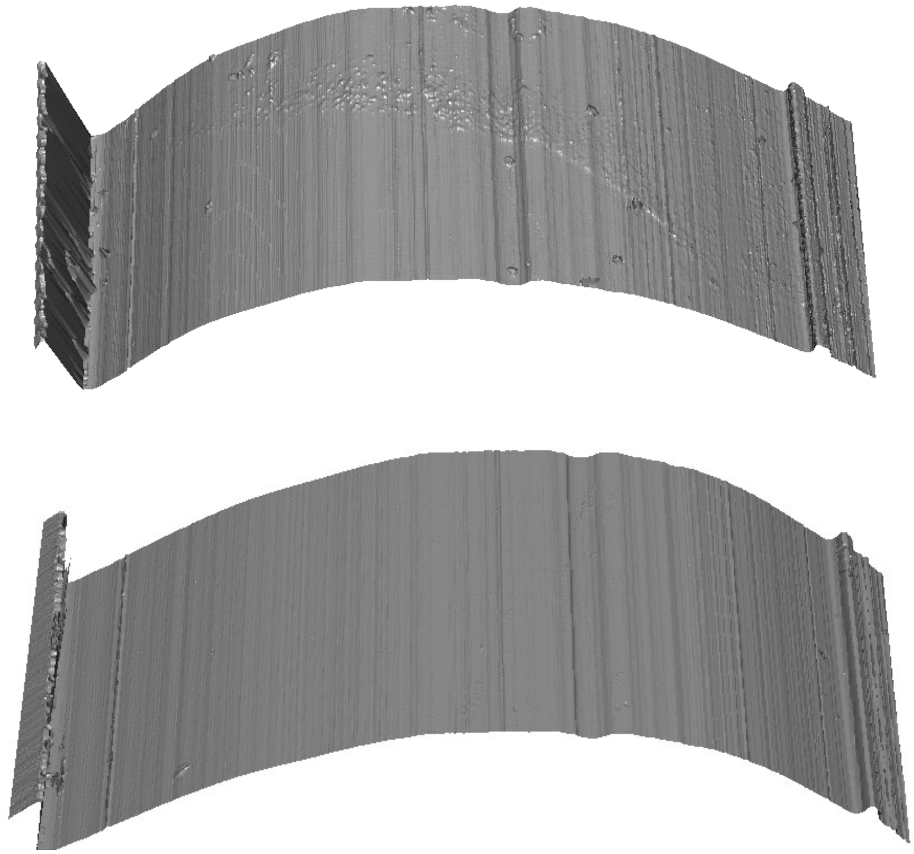


Nanofocus μScan Confocal Microscope for measurement of surface texture, and especially the topography of bullets and casings

coherence scanning Interferometry, and confocal chromatic probing, will be developed. As a specific application of optical techniques, the project will continue to evolve the physical standard, SRM 2460, for optical examination of bullets and an analogous standard, SRM 2461, for examination of the casings. This work has application to the infrastructural work on optical techniques, discussed above, because the similarity between surface topography as measured by stylus and optical techniques is of crucial importance to establishing the validity of optical techniques.

Impacts:

- The NIST Report, Surface Topography Analysis for a Feasibility Assessment of a National Ballistics Imaging Database, was heavily cited by the National Academies in their publication, entitled Ballistic Imaging, one of the reports leading to a substantial ongoing re-evaluation of the use of firearms evidence from fired bullets and casings.
- The On-line Surface Metrology Algorithm Testing System has received thousands of hits by users and has been used in an international comparison of Surface Metrology Software, recently published by NPL (UK). The NIST results compare very well with those of other national laboratories, NPL and PTB, and were useful in assessing the weaknesses of three commercial software packages, which were also compared in the study.
- The NIST random profile roughness specimens are electro-formed replicas from master specimens produced by John Song in 1985. Six sets of the replica specimens were calibrated by NIST in 2004 under an MEL Director's Reserve Project and have been available for loan to US companies. The companies that have taken advantage of this service include GE Aviation, Cummins Engine, InsituTec. Inc., Timken Company, REM Chemicals Inc and Woodward Engineering. NIST has received very good feedback on this program. For example, in April, 2009, a Design Engineer of GE Aviation sent an email to NIST stating: "These NIST calibration specimens gave GE engineering confidence that we could measure surface finishes down in the single digit micro-inch Ra range, and also allowed us to verify that we could measure the surface finish on the same part at 3 different locations and report the same result at all locations. The NIST specimens allowed GE engineering to expand our measurement capabilities and eliminate gauge R&R issues between various manufacturing shops, and I would like to say thanks to NIST for providing these specimens as they were invaluable for helping us to clear up surface finish measurement issues we were having at the time."

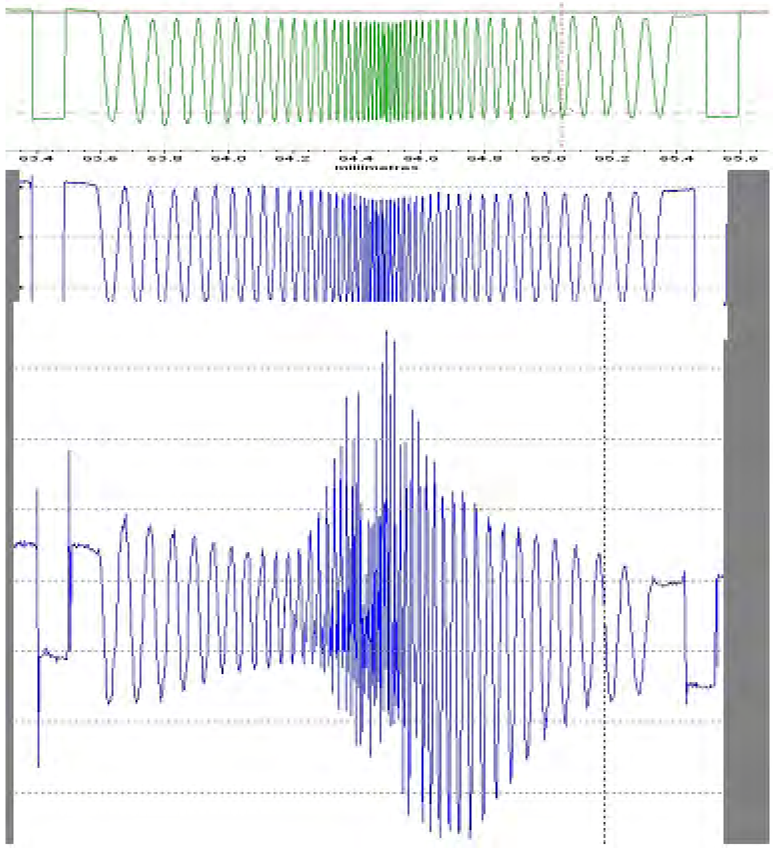


Topographic images of a polymer replica (top) of a standard bullet and an original standard bullet (bottom) at one land engraved area.The replica was fabricated by the BKA, the equivalent agency in Germany of the FBI in the U.S.

- In 1995, PED developed a Microform Calibration System for calibration of the standard Rockwell diamond indenters. This system was internationally recognized as the calibration system with the lowest uncertainty in the world. Using this system, two NIST Primary Rockwell Indenters were established, by which hundreds SRM standard hardness blocks were calibrated for implementation of the US National Rockwell hardness (HR)Scales. In 2009, this system was upgraded and automated so that the for a Rockwell diamond indenter drop from hours to only 20 minutes. The new results from this tool have demonstrated the long term stability of both the NIST Primary Rockwell Indenter and the NIST Microform Calibration System. Both are important issues for maintaining the US Rockwell hardness scales and for the establishment of a worldwide unified HR scale.

Accomplishments:

- First systematic observation of large biases in roughness measurement by coherence scanning interferometric microscopes in the 50-200 nm range of roughness amplitude and publication of the results in Applied Optics (2005) and the International Journal of Advanced Manufacturing Technology (2007),
- Participation in The SIM 4.8 international comparison of roughness and step height measurements and publication of the results in Metrologia (2006),
- Completion of the National Institute of Justice / NIST Office of Law Enforcement Standards Project on “Surface Topography Analysis for a Feasibility Assessment of a National Ballistics Imaging Database” and publication of the report as NISTIR 7362 (2007),
- Development of the On-line Surface Metrology Algorithm Testing System and publication of the report in Precision Engineering (2007); this is currently the only on-line system with calculations



Recent results measured on a new chirped roughness specimen currently under development at PTB. The stylus profile (top) agrees well with the profile from the confocal microscope obtained with a 50X objective (middle), but the data for a 10X objective are clearly distorted especially when measuring the shorter spatial wavelengths. The height of the surface structure is about 1 µm and the length of the profile is about 2 mm. The longest spatial wavelengths are about 100 µm and the shortest are about 10 µm.

of 3D roughness parameters and calculations of uncertainties of roughness parameters,

- Leading ISO Technical Committee 213/ Working Group 16/ Project Team on Optical Methods. This team has produced six draft standards for 3D measurement of surface texture with optical techniques, which are individual parts under the umbrella of ISO 25178: Geometrical product specification (GPS) — Surface texture : Area l:
- Final Draft International Standard (FDIS) Part 6: Classification of methods for measuring surface texture,
- FDIS Part 602: Nominal characteristics of non-contact (confocal chromatic probe) instruments,
- DIS Part 603: Nominal characteristics of non-contact (phase shifting interferometer) instruments,
- DIS Part 604: Nominal characteristics of non-contact (coherence scanning interferometry) instruments,
- Committee Draft Part 606: Nominal characteristics of non-contact (focus variation) instruments,
- Working Draft Part 60X: Nominal characteristics of non-contact (imaging confocal) instruments.
- Acceptance and topography measurement of 150 units of SRM 2461, Standard Casings, and dissemination to 19 firearms experts under the National Ballistics Imaging Comparison Project; successful completion of two workshops under the Project.

Publications List:

- “NIST Bullet Signature Measurement System for RM (Reference Material) 8240 Standard Bullets,” L. Ma, J. Song, E. Whitenton, A. Zheng, T.V. Vorburger, and J. Zhou, J. Forensic. Sci. 49, 649 (2004).
- “Internet-based Surface Metrology Algorithm Testing System,” S. Bui, T. Renegar, T. Vorburger, J. Raja, and M. Malburg, Wear 257, 1213 (2004).
- “Virtual Surface Calibration Database,” S. Bui, T. Renegar, and T. Vorburger; XI Int’l Colloquim on

"These NIST calibration specimens gave GE engineering confidence that we could measure surface finishes down in the single digit micro-inch Ra range, and also allowed us to verify that we could measure the surface finish on the same part at 3 different locations and report the same result at all locations. The NIST specimens allowed GE engineering to expand our measurement capabilities and eliminate gauge R&R issues between various manufacturing shops, and I would like to say thanks to NIST for providing these specimens as they were invaluable for helping us to clear up surface finish measurement issues we were having at the time." ...GE Aviation

Surfaces, Prod ed M. Dietzsch (Shaker Verlag, Aachen) (2004) Part II, pp. 93-101.

- "Virtual Surface Calibration and Computational Uncertainty," S. Bui, T. Renegar, and T. Vorburger, Proc. Amer. Soc. Prec. Eng. (2004).
- "Standard Deviation or Standard Deviation of the Mean – How to Report Statistical Variation in Surface Calibrations?," J. Song and T. Vorburger Proc. Amer. Soc. Prec. Eng. (2004).
- "Discrepancies between Roughness Measurements Obtained with Phase-shifting and White-light Interferometry," H. Rhee, T.V. Vorburger, J. Lee, and J. Fu, Appl. Opt. 44, 5919 (2005).
- "2D and 3D Surface Texture Comparisons Using Autocorrelation Functions," J. Song, L. Ma, E. Whitenton, and T. Vorburger, Key E. Song, Engineering Materials 295-296, 437 (2005)
- "A Metric for the Comparison of Surface Topographies of Standard Reference Material (SRM) Bullets and Casings," J.F. Song, T. Vorburger, L. Ma, J.M. Libert, S.M. Ballou, Amer. Soc. Prec. Eng. (October, 2005).
- "Correlation of Topography Measurements of NIST SRM 2460 Standards Bullets by Four Techniques," J. Song, T. Vorburger, H. Rhee, A. Zheng, L. Ma, J. Libert, S. Ballou, B. Bachrach, and K. Bogert, Meas. Sci. Technol. 17, 500 (2006).
- "International Comparison of Surface Roughness and Step Height (Depth) Standards, SIM 4.8," K. Doytchinov, F. Kornblit, C. Colin Castellanos, J.C.V. Oliveira, T. Renegar, and T.V. Vorburger, Metrologia 43 04002 (2006) and http://www.bipm.org/utils/common/pdf/final_reports/L/S2/SIM.L-S2.pdf.
- "The Effect of Gaussian Filter Long Wavelength Cutoff μc in Topography Measurements and Comparisons," J. Song, L. Ma, T.V. Vorburger, Proc. Amer. Soc. Prec. Eng. (2006).
- Surface Topography Analysis for a Feasibility Assessment of a National Ballistics Imaging Database, NISTIR 7362, T. Vorburger, J. Yen, B. Bachrach, T.B. Renegar, J.J. Filliben, L. Ma, H.-G. Rhee, A. Zheng, J.-F. Song, M. Riley, C.D. Foreman, S.M. Ballou (National Institute of Standards and Technology, Gaithersburg MD, 2007).
- "Comparison of Optical and Stylus Methods for Measurement of Rough Surfaces," T.V. Vorburger, H.-G. Rhee, T.B. Renegar, J.-F. Song, and A. Zheng, Int. J. Adv. Manuf. Technol. 33, 110 (2007), DOI 10.1007/s00170-007-0953-8.
- "Surface Metrology Algorithm Testing System," S.H. Bui and T.V. Vorburger, Prec. Eng. 31, 218-225 (2007).
- "Topography Measurements and Applications," J.-

F. Song and T. Vorburger, Proc. SPIE 6280, 62801T (2006); doi: 10.1117/12.716162.

- "A Novel Parameter Proposed for 2D and 3D Topography Comparisons," J. Song and T. Vorburger, Proc. SPIE 6672, 66720M (2007); doi:10.1117/12.734424
- "Verifying Measurement Uncertainty Using a Control Chart with Dynamic Control Limits," J. Song and T. Vorburger, Measure 2, 76-80 (2007).
- "NIST Standard Bullets and Casings Project," J. Song, T. Vorburger, S. Ballou, Proc. Amer. Soc. Prec. Eng. 2007 (Amer. Soc. Prec. Eng., Raleigh, NC, 2007).
- "Topography measurements for determining the decay factors in surface replication,"
- J. Song, P. Rubert, A. Zheng and T.V. Vorburger. Meas. Sci. Technol. 19 (2008) 084005; doi:10.1088/0957-0233/19/8/084005.
- "Optimizing a Gaussian Filter Long Wavelength Cutoff for Improving 3D Ballistics Signature Correlations," J. Song, L. Ma, T. Vorburger, and S. Ballou, Proc. Amer. Soc. Prec. Eng. (2008).
- "Traceability for Ballistics Signature Measurements in Forensic Science," J. Song, T. Vorburger, S. Ballou, T. Renegar, A. Zheng, and M. Ols, IMEKO Workshop on Traceability to support CIPM MRA and other international arrangements (Torino, November 6-7, 2008) and Journal of Measurement, DOI 10.1016/j.measurement.2009.07.006. http://dx.doi.org/10.1016/j.measurement.2009.07.006.
- "Optimization of Cellular Response on Flexible Surfaces Using Chemically Bound Proteins," L.M. Pakstis, J.P. Dunkers, A. Zheng, T.V. Vorburger, T.P. Quinn, and M.T. Cicerone, J. Biomed. Mat'ls. Res. A (in press).
- "Pilot Study of Automated Bullet Signature Identification Based on Topography Measurements and Correlations," W. Chu, J. Song, and T. Vorburger, J. Forensic Sci. (in press).
- "Improved Firing Pin Signature Correlations by Optimizing Gaussian Regression Filter", L. Ma, J. Song, T. Vorburger, and S. Ballou, J. Forensic Sci. (in press).
- "Three Steps towards Metrological Traceability for Ballistics Signature Measurements" Song, J., Vorburger, T., Ballou, S., Ma, L., Renegar, T., Zheng, A., Ola, M., in Proceedings of the ISMTII 2009, Saint-Petersburg, June, 2009.
- "Striation Density for Determining the Identifiability of Bullets," W. Chu, J. Song, T. Vorburger, and S. Ballou, J. Forensic Sci. (in press).
- "NIST SRM (Standard Reference Material) 2460/2461 Standard Bullets and Casings Project," Song, J., Vorburger, T., Renegar, T., Zheng, A., Thomson, R., Silver, R., Ols. M., to be published in the proceedings of 4th International Conference on Forensic Weapons Study (ICFWS 2009), Saratov, Russia, October 13-14, 2009.
- "Geometric measurement comparisons for Rockwell diamond indenters," Song, J., Low, S., Zheng, A., in the Proceedings of the IMEKO XIX World Congress, September, 2009, Lisbon, Portugal.

Presentations:

Keynote

- Song, J.F. "Three Steps towards Metrological Traceability for Ballistics Signature Measurements," Proceedings of the ISMTII 2009, Saint-Petersburg, June, 2009.

Invited

- Vorburger, T.V. "Standard Bullets and Casings for Ballistics Inspection Laboratories",
- 15TH ENFSI Eexpert Working Group on Firearms Meeting, Dubrovnik, Croatia October 14-17, 2008
- Vorburger, T.V. "The National Nanotechnology Initiative and Nano-scale Length Metrology,"
- Simposio de Metrologia, Querétaro, Mexico, October 22, 2008.
- Vorburger, T.V. "Topics in Surface and Nano Metrology Related to Biotechnology," Surface Textures for Bioengineering Seminar, Worcester Polytechnic Institute, Worcester, MA, 11/13/08.
- Song, J.F. "Traceability for Ballistics Signature Measurements in Forensic Science," IMEKO Workshop on Traceability to support CIPM MRA and other international arrangements, Torino, Italy, November 6-7, 2008
- Vorburger, T. V. "Nano- and Atomic-scale Length Metrology," Singapore Institute of Manufacturing Technology, Singapore, 11/20/08.
- Vorburger, T. V. "Introduction to Surface Finish Metrology," Singapore Institute of Manufacturing Technology, Singapore, 11/20/08.
- Vorburger, T. V. "Comparisons of Methods for Measurement of Surface Finish," Singapore Institute of

Manufacturing Technology, Singapore, 11/20/08.

- Vorburger, T.V. "ISO Standards (for Surface Texture) Update,", ASME B46 Surface Quality Seminar, Romulus, MI, April 24, 2009.
- Song, J.F. "Geometric measurement comparisons for Rockwell diamond indenters," in Proceedings of the IMEKO XIX World Congress, September, 2009, Lisbon, Portugal.

Contributed

- Vorburger, T.V. "Applications of Cross-Correlation Functions," 12th International Conference on the Metrology and Properties of Engineering Surfaces, Rzeszow, Poland, July 8-10, 2009

Workshops:

- NIST led Workshop, National Integrated Ballistics Information Network (NIBIN) User Congress, National Ballistics Imaging Comparison (NBIC) Workshop, January 8, 2009.

SRM/RMs Produced:

- SRM 2460, Standard Bullets (2007)
- SRMs 2073a, 2074, 2075, Recertified (2009).

Recognitions:

- T. Vorburger, Co-Chair of ASME Manufacturing Science and Engineering Conference Symposium on New Developments in Nanofabrication, Nanometrology, and Applications, Evanston, IL, October 2008 and West Lafayette, IN, October, 2009.

Customers:

- U.S. customers national and local forensic laboratories such as the National Laboratory Center of ATF, and the Central Laboratory of FBI; the local ATF, FBI and police laboratories; the NIBIN users including 250 forensic laboratories nationwide;
- International customers include the BKA (Federal Criminal Police Office) in Germany, the FTI (Forensic Technology Inc.) in Canada and a private company (Spzoocenzin Co.) in Poland. With the increase of using ISO 17025 standard in the international forensic community, more international customers are expected in the near future.
- Metrology suppliers to the automotive, aircraft, and MEMS industries

Collaborators:

- Collaborators include ATF, FBI, BKA, NIBIN, Forensic Technology Inc. Canada, Rubert Co. Ltd. in UK, and the ASME B46 Committee on the Classification and Designation of Surface Qualities;

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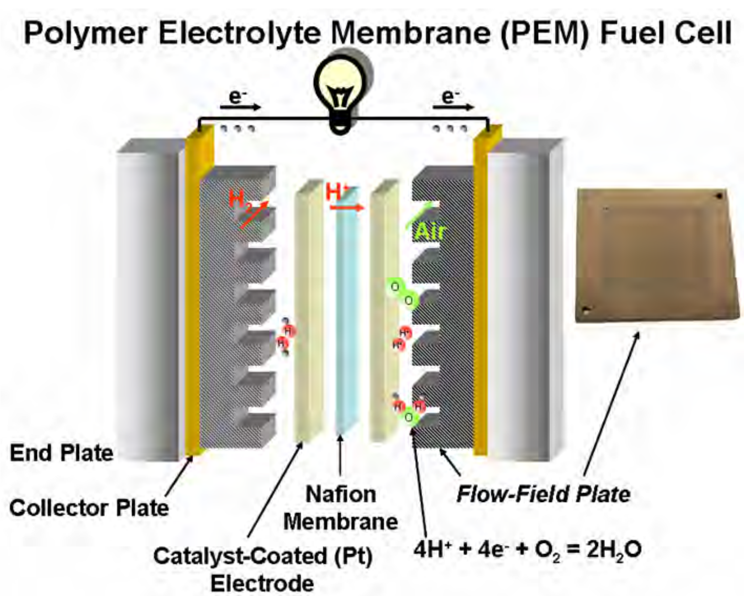
Precision Engineering Division Cross Agency Collaboration:

Metrology for Fuel Cell Manufacturing

The Precision Engineering Division has a long history of working in collaboration with other Federal Government Agencies to develop mission specific metrology. One example of such ongoing work is the state-of-the-art research with the Department of Energy (DOE) Hydrogen Program to conduct a research effort, over a multi-year period, addressing fuel cell manufacturability issues. Specifically, this effort focuses on bipolar plates (also known as flow field plates), membrane electrode assemblies (MEAs), and fuel cell stacks as shown in the adjacent figure. Each of the three PED Programs and the Manufacturing Metrology Division (MMD) of MEL are contributing to the success of this work.



Background: The primary goal of the DOE's Hydrogen Program manufacturing research plan is a roadmap to reduce the cost of manufacturing hydrogen and fuel cell technologies (http://www.hydrogen.energy.gov/manufacturing.html). This roadmap includes analysis and evaluation of fabrication and assemby of critical fuel cell components, including bipolar plates and MEAs. Bipolar plates need high precision fabrication and measurement methods, including high-speed in-process metrology, to ensure the durability and performance needed in fuel cell systems. These components must also be assembled as cells and into stacks in reliable, timely ways that help ensure both the functionality of the fuel cell system and the overall cost-effectiveness of the production operations. This manufacturing research by the DOE's Hydrogen Program has been authorized by the Energy Policy Act of 2005, Section 805 (e)(6).

NIST/PED in close collaboration with the MMD is contributing to this through research of the technologies employed in fuel cell manufacturing by addressing fundamental measurement and manufacturing issues associated with bipolar plates and MEAs. NIST is creating a base of publicly available, pre-competitive knowledge to understand the relationships between fuel cell system performance and manufacturing process parameters and variability. Such understanding will play a major role in fuel cell design, tolerances, and specifications, and is integral to implementing design for manufacturability.

Through this collaborative work, DOE and NIST will further the Nation's understanding of the basic metrology and manufacturing technologies necessary to enable the transition to cost-effective and predictable, high-volume fuel cell production. NIST will utilize its state-of-the-art measurement, manufacturing facilities and equipment, operating in close collaboration with the U.S. industrial and academic fuel cell community. DOE is providing NIST with overall strategic input to help ensure that the NIST research efforts are aligned with DOE programmatic needs and directions. This effort is currently divided in the three subprojects:

1. Cause-and-Effect: Flow Field Plate Manufacturing Variability and its Impact on Performance. The objective of this subproject is the development of publicly available engineering data quantifying the correlation between flow field plate

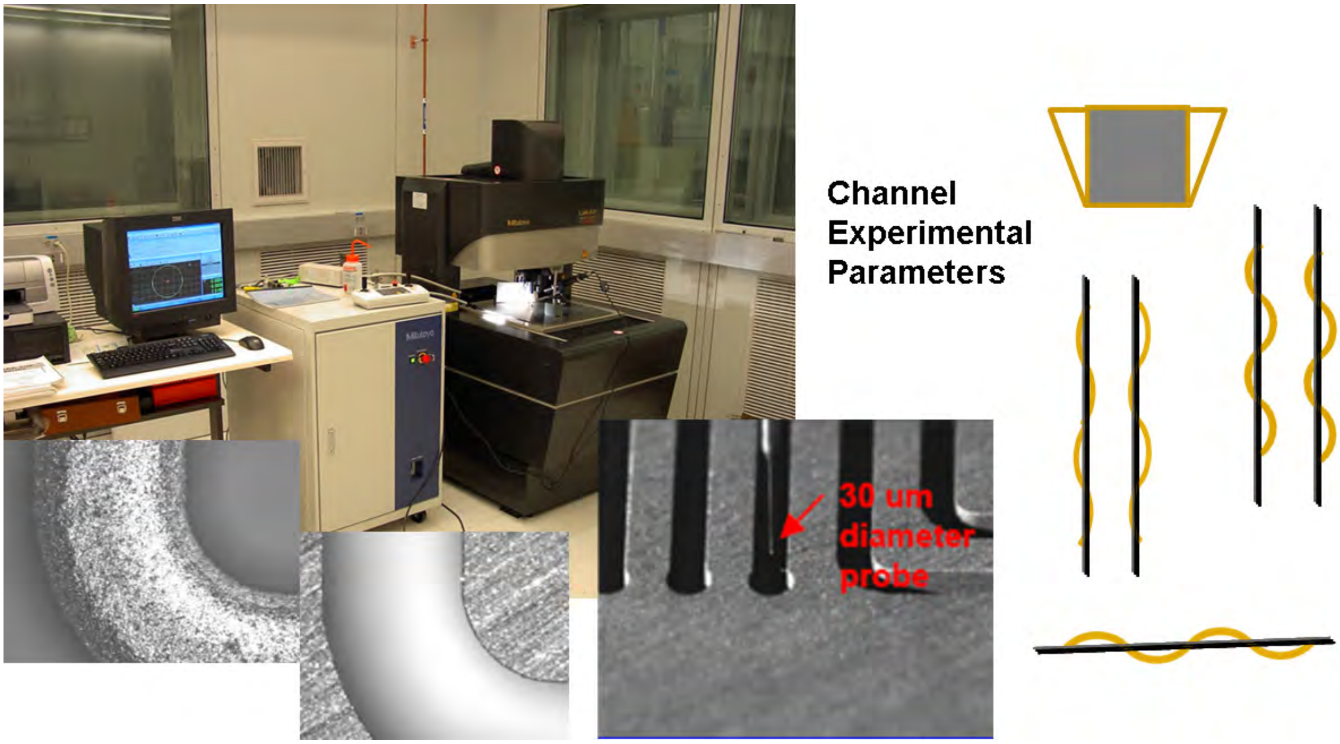


Figure 2. Reference Metrology and Experiment Plate Parameter Verification using a dual-probe micro-feature coordinate measuring machine.

manufacturing variability and fuel cell performance. For this project the single cell fuel cell performance testing capability at NIST has been developed (Figure 2) and redundantly verified and validated. The design of the experiment has been determined, the geometric fuel cell parameters to be varied have been selected (i.e.,variation in channel side wall and bottom straightness, side wall taper, and variation-in-channel width). In addition, the experimental flow field plates have been fabricated, dimensionally characterized (Figure 2), and are ready for assembly in the reference single cell fuel cell. Continued work will focus on developing a performance testing protocol that minimizes variability due factors other than those introduced by the geometric perturbations of the experimental plates and demonstrates stability (or at least accurately predictable instability) over the time period of the experimental plate testing. Due to unanticipated complications regarding the needed continuous unattended operation of the NIST Fuel Cell Testing Laboratory to minimize variability associated with start-up and shut-down of the fuel cell during performance testing, NIST recently decided to call upon our colleagues at Los Alamos National Laboratory (LANL) to perform the performance testing activity to ensure a timely project completion. Currently this project is slated for completion by the end of FY2010 and with LANL's assistance we are still on target to meet this goal.

2. Non-Contact Sensor Evaluation for Bipolar Plate Manufacturing Process Control and Smart Assembly of Fuel Cell Stacks. The objective of subproject 2 is the development and/or evaluation of high-speed non-contact sensors for application in process control of bipolar plates (Figure 3). The evaluation (Figure 4) will include: suitability

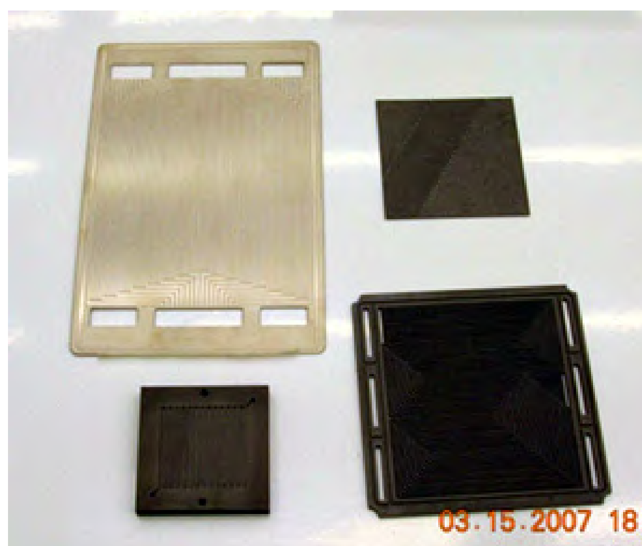


Figure 3. Common Bipolar Plates Used in PEM Fuel Cells Representing Different Materials (Carbon Composite & Coated Metallic) and Different Manufacturing Methods.

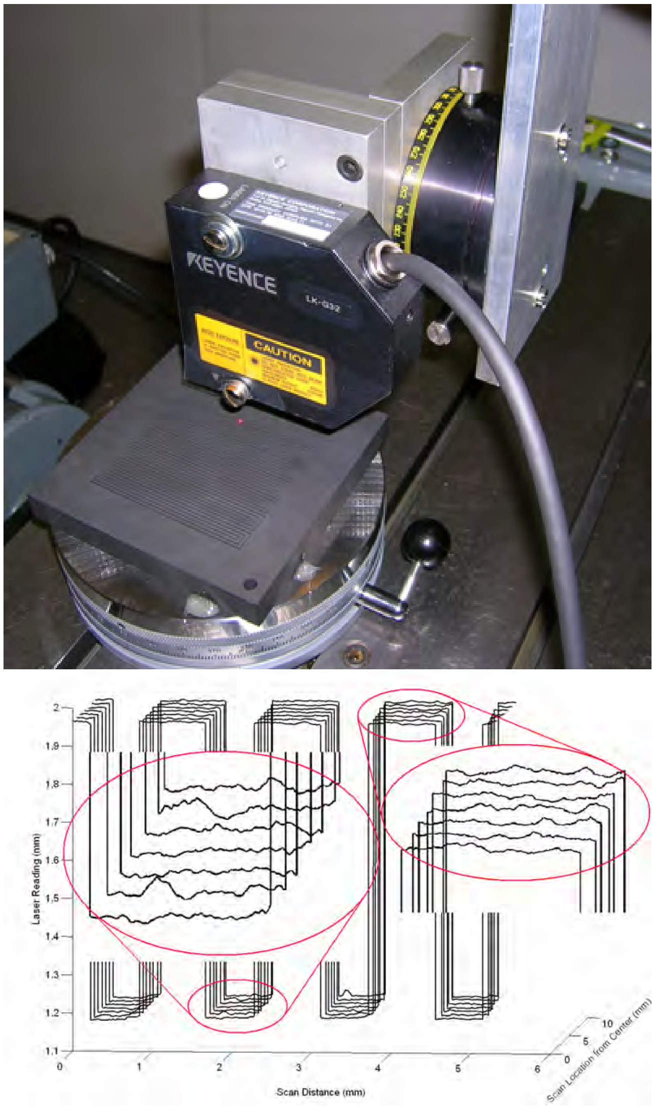


Figure 4. Laser spot triangulation probe evaluation; (upper) sensor testbed, (lower) graphical representation of sensor measurements showing feature size and form.

based on typical plate materials and methods of fabrication, dimensional parameters of interest (i.e., channel depth, channel width, channel spacing, location of features, and plate parallelism and flatness), development of measurement protocols, accuracy evaluation as a function of scan speed, and approaches to achieving contractual traceability requirements. Further, demonstrated sensor technologies will be integrated into a system that uses measurement information of individual plate parallelism to enable smart assembly to achieve tighter overall stack parallelism tolerances while reducing individual plate parallelism tolerances.

3. Optical Scatterfield Microscopy for Online Catalyst Coating Inspection of Proton Exchange Membrane (PEM) Fuel Cell Soft Goods. Subproject 3 conducts research into the application of optical scatterfield microscopy (OSM) as an in-situ, non-destructive, high-speed, and far less-expensive measurement alternative to current, typically ex-situ, measurement technologies (i.e., Scanning Electron Microscopy (SEM) or Transmission Electron Microscopy (TEM)) or X-ray Flourescence Spectroscopy (XRF). SEM and TEM are typically used to characterize the numerous parameters of interest. The parameters of interest include, but are not limited to the following: mean platinum size and distribution, platinum loading, defect size and distribution, platinum agglomeration, and homogeneity. This new measurement technique was originally developed by NIST /PED researchers for process control of discrete structures arranged in predetermined patterns commonly found in the semiconductor industry. For this research application, the technique will be tested on similar sized randomly oriented particles which commonly platinum in a carbon/ionomer matrix (Figure 5). Application will involve developing protocols and comparable quantitative measures using traditional catalyst inspection techniques. Evaluation of the sensitivity of the scatterfield mocrroscopy technique to the different parameters and development of image analysis algorithms are in progress (Figure 6). Potentially tool design modifications to optimize the sensitivity of the technique to those parameters of interest are also being

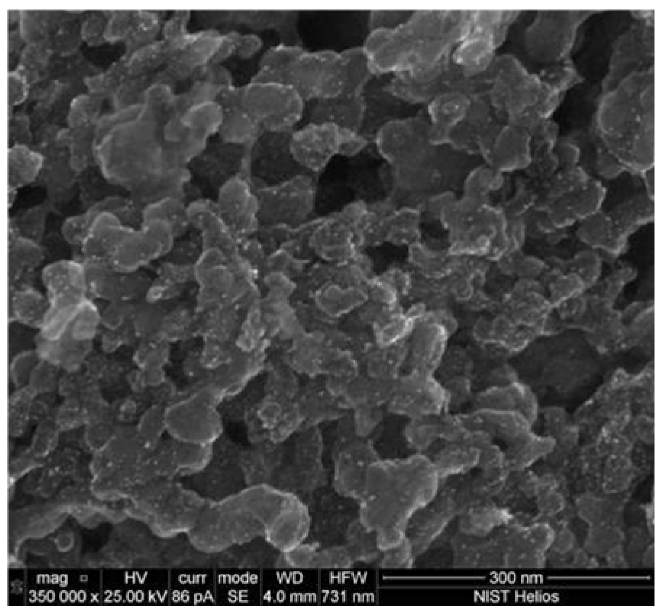


Figure 5. SEM image of a common platinum catalyst coated membrane (CCM) showing very small platinum particles dispersed on larger carbon particles in an ionomer matrix.

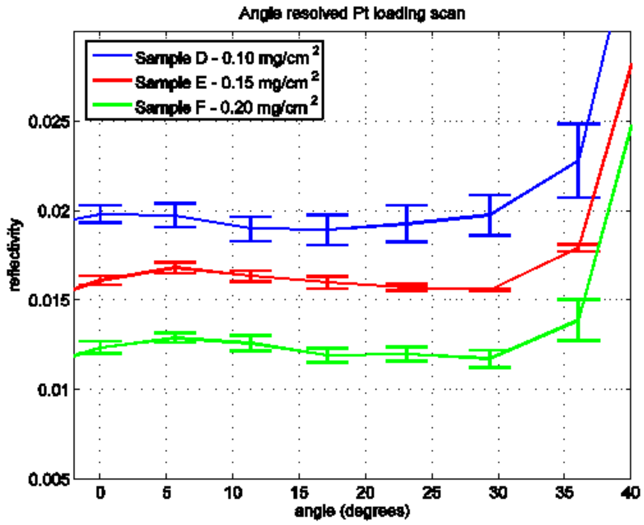


Figure 6. Preliminary Optical Scatterfield Microscopy results showing sensitivity to platinum loading on a catalyst coated membrane.

evaluated. This work requires close cooperation with MEA manufacturers to ensure the parameters of interest are and remain relevant to the process and that samples tested represent the latest developments in industry. The scope of this project work will be executed in cooperation with the National Renewable Energy Laboratory (NREL) who has a complimentary effort in fuel cell manufacturing research and development. Collaboration efforts are currently underway to finalize necessary agreements so that NREL and NIST can freely share information from industry partners between organizations. Laboratory (NREL) who has a complimentary effort.

Collaborators:

- Los Alamos National Laboratory (http://www.lanl.gov/orgs/mpa/mpa11/fuelcell.htm).
- National Renewable Energy Laboratory (http://www.nrel.gov/hydrogen/proj_manufacturing.html).
- Various Fuel cell MEA and bipolar plate manufacturers

Publications/Presentations:

- DOE 2008 Annual Merit Review Presentations, June 13, 2008. (http://www.hydrogen.energy.gov/pdfs/review08/mf_7_stanfield.pdf)
- DOE Hydrogen Program 2008 Annual Progress Report, Sec. 6, Technical Report No 3 (2008) (http://www.hydrogen.energy.gov/pdfs/progress08/vi_3_stanfield.pdf).
- Silver, R. M., B.M. Barnes, R. Attota, J. Jun, M. Stocker, E. Marx, and H. Patrick, "Scatterfield Microscopy for Extended Limits of Image-Based Optical Metrology," Applied Optics, Vol. 46, No. 20 (2004).

Related Publications:

- NRELTechnical Report NREL/TP-560-416552007, (2008)Status of Manufacturing: Polymer Electrolyte Membrane (PEM) Fuel Cells (http://www.nrel.gov/hydrogen/pdfs/41655.pdf).

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Nanomanufacturing Metrology Program – *Enabling accuracy for nanotechnology manufacturing*

Program Manager: Dr. Richard Silver

Challenge:

The increasing pace of technological change in nanomanufacturing makes accurate dimensional metrology critical to innovation and realization of quality products. Industries currently manufacturing at the nanoscale are challenged by the need for infrastructural metrology and standards to enable process control, enhance product quality and to bring innovative new products to market. The goal is to advance U.S. leadership in nanomanufacturing through the development of physical standards traceable to the International System of Units (SI) and through the development of physics-based models and calibration techniques which enable accurate determination of dimensional information.

Overview:

The Nanomanufacturing Metrology (NanoMet) Program focuses on the development of solutions to dimensional metrology needs of the nanomanufacturing industry by providing dimensional standards, calibrations and infrastructural metrology for measurements in the nanoscale having subnanometer precision. Semiconductor manufacturing, data storage, and photonics industries are currently the main customers. The semiconductor manufacturing industry is a major current focus with critical dimensions of mass produced product in the <50 nm region today. The program remains at the forefront of rapidly evolving metrology needs through active interaction with industry leaders and industry consortia (such as SEMATECH); and by strong participation and leadership in industrial roadmaps (such as the International Technology Roadmap for Semiconductors). The program also serves other industries (e.g., flat panel display or microelectromechanical (MEMS) fabrication), government agencies (e.g., DARPA or Department of Energy Labs), and academic researchers that need traceable dimensional metrology at the sub-micrometer scale. The goal is to provide the calibration techniques and artifacts which realize the Système International d'unités (SI) definition of the meter to meet the most demanding nanoscale industrial needs.

The NanoMet Program implements its strategy through a series of objectives, each containing a set of time-sequenced projects. The projects in these objectives will create and deliver solutions to significant dimensional metrology problems, typically leading to new calibrations or standard reference materials.

The NanoMet Program continually evaluates new metrology opportunities to assist industry and reviews existing projects for consistency with program objectives. An ongoing goal is to refine calibration methods and standard artifacts in order to reduce the uncertainty in the realization of the SI definition of the meter as it is supplied to industry. This project relies on continual improvement in instrument performance and consistency of measurements between instruments and methods. In addition the work defined within this program addresses several of the identified and validated U. S. Measurement System Measurement Needs (MN). The specific MNs and page numbers from Appendix B of the Report are found at the end of this document.

Why NIST?

Nanomanufacturing represents one of the crucial markets for advanced manufacturing and innovative product development. Accuracy and precision have become essential to nanotechnology manufacturing. Requirements for measurement standards, instrumentation and standardized calibration techniques are acute when manufacturing in the nanometer domain. NIST has extensive expertise in dimensional metrology at the nanometer scale. NIST has been working with nanomanufacturing industries, such as leading edge semiconductor or MEMs device manufacturers, and has been integrally involved in the development of standards, instrumentation, calibration methods, and measurement science for the advancement of nanotechnology manufacturing. This is a manufacturing arena in which NIST uniquely possesses the expertise and instrumentation to facilitate U.S. leadership.

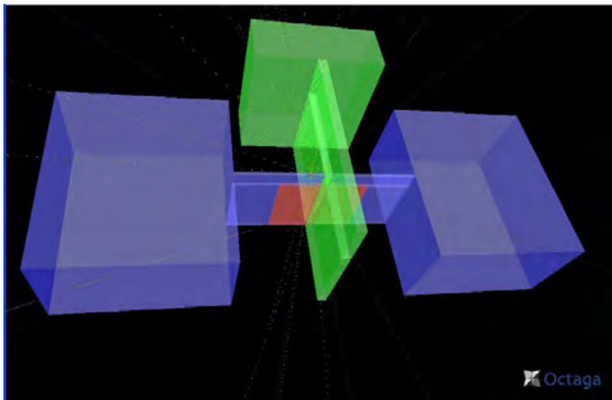
Program Objectives:

Objective 1: Provide industry with accurate and timely dimensional scale metrology at the nanoscale to enhance U.S. productivity and innovation

- Fundamental to this objective is meeting the current and anticipated one and two dimensional scale calibration and the SI dimensional traceability requirements for improved yield and product qualification for the constantly evolving nanomanufacturing industry. The fundamental basis for length metrology is traceability to the definition of the meter in the International System of Units (SI). The challenge is to realize this definition in practice through qualified metrology instruments and calibrated standards and reference materials that form the basis for dissemination.
- The goals for this objective are to provide accessibility to the SI unit of length to the nanomanufacturing industries, such as semiconductor manufacturing, data storage, and photonics, by means of reference metrology instruments and calibration standards that are compatible with industry metrology instrumentation. This is required for the industry to remain on the technology roadmaps as device structures are reduced to less than 18 nm within the next 10 years. These nanometer scale manufacturing requirements drive push the limits of length scale measurement. This program identifies the current limits to length scale measurement as shown by uncertainty budgets and develops new technology to reduce them.

Objective 2: Provide accurate critical dimension (linewidth metrology) traceable to the meter.

- Significant manufacturing metrology challenges exist beyond scale calibration for the accurate determination of the size width and length of physical features. Measurement of linewidth or critical dimension (CD) continues to be one of the most fundamental dimensional metrology needs in the semiconductor and nanomanufacturing industries. Semiconductor manufacturers refer to this continually decreasing measurement limit as critical dimension (CD) metrology. The critical dimension size and tolerance decreases as technology progresses. The demand is so complex and ubiquitous that no single metrology technique can provide the entire solution. Three major techniques will be used within the linewidth metrology objective: 1) critical-dimension AFM (CD-AFM), 2) SEM, and 3) optical microscopy. At this scale, the largest component of measurement uncertainty is usually associated with the interaction of the probe (e.g., mechanical



stylus, photons, or a beam of charged particles) with the specimen. The principal challenge in linewidth metrology is to accurately define the position of the physical edge of a feature within the metrology instrument response profile. Each instrument exhibits its own characteristic response profile due to the bandwidth of the electronics, signal collected or probe used. The measurand also contributes to the response as well. The result is an increased linewidth measurement uncertainty. This uncertainty can easily become an undesirable value when dealing with nanometer structures and NIST is continually striving to decrease this measurement limitation.

- In order to reduce linewidth measurement uncertainty, new measurement techniques producing sharper edge profiles will be developed and adopted. But, ultimately no method has been able to fully achieve the required resolution, and it becomes necessary to assign the physical edge to a definite position within the broadened signal. This in turn requires understanding and modeling the physical process that produce the broadening so that it can be compensated, and as a result electron beam, scanned probe and optical modeling have become major components of this project.

- NIST expertise will focus on improving the effectiveness of the instrument response profile by using shorter-wavelength light in optical microscopy (OM); smaller beam focus spots in scanning electron microscopy (SEM); or sharper, better characterized tips in CD atomic force microscopy (CD-AFM). Such improvements are required to advance this metrology. This contribution to the measurement uncertainty is a greater fraction of the total as feature sizes decrease below 50 nm. Consequently, physics-based modeling is required to understand and overcome this challenge. Validated models for each of the measurement techniques are critical to accurate measurements. In addition, experimental intercomparisons between the various measurement methods are required to further validate the models. To provide traceability, NanoMet will develop custom reference measurement instruments (SEM, SPM and OM) which have been highly engineered to provide traceability through the incorporation of the most accurate laser interferometry. Standard Reference Material (SRM) standards will be certified with these reference measurement instruments to accurately calibrate production instrumentation.

Objective 3: Provide accurate overlay and registration metrology with subnanometer accuracy to enable manufacturing of the most advanced, fastest semiconductor devices.

- Developing advanced position metrology, techniques and standards for overlay and registration are critical to the semiconductor industry and will become more important for other nanomanufacturing in the future. The relative overlay of features from different manufacturing process levels is considered one of the most demanding measurement requirements due to the direct effects on device performance. NIST will develop infrastructural metrology for the accurate placement (registration) of multiple layers with sub-nanometer accuracy. Since optical techniques are often best suited to these tasks, research into novel optical overlay and registration instrumentation and target structures is essential. In semiconductor applications, development of new instrument innovations and target designs will be jointly developed with the leading industrial consortium, SEMATECH. These structures enable improved overlay resolution in a smaller, in-chip format of sub-resolution features. It is critical to develop high-resolution optical overlay techniques that are extensible for several manufacturing generations. The progression of technology demands that new generations of SRMs meeting new production challenges must continually be developed to ensure the progress of the industry.

- NIST will work jointly with the leading industrial consortium, SEMATECH, to provide improved overlay instrument resolution in a smaller, in-chip format of sub-resolution features that are extensible for a number of manufacturing generations. SRMs for overlay calibration will also be designed and fabricated. The collaborative implementation of new target designs, instrument optimization and modeling, and calibration techniques includes new reticle design and wafer fabrication in the continuing effort undertaken jointly with key industrial partners.

- The overlay metrology project is an internationally recognized effort with the goal of developing techniques and targets for improved overlay metrology, primarily for the semiconductor industry. The project has relied on close collaboration with industry leaders in optical tool development and users of overlay metrology tool sets as well as with SEMATECH. NIST will continue to work with the industrial partners to ensure the progress of the industry

The Nanomanufacturing Metrology Program accomplishes these objectives through 7 projects:

- Optical Linescale Metrology
- Nanometer Scale Dimensional Metrology
- Wafer Level AFM Metrology for Critical Dimension Measurements
- Wafer Level SEM Metrology for Critical Dimension Measurements
- Wafer Level SEM Metrology for Critical Dimension Measurements: SEM Modeling
- Photomask Dimensional Metrology
- Overlay Instrument and Wafer Target Designs

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Optical Linescale Metrology

Industry Need:

The cornerstone of traceability for the SI Unit of length (the meter) is the Linescale Interferometer (LSI) which provides world-class one-dimensional scale calibrations traceable to the SI. The LSI performance is verified by international intercomparisons, where it consistently performs as one of the top instruments in the world. This facility provides calibrations for customers in the nanomanufacturing industry, such as major metrology tool suppliers. It is also the traceability link for most of the other dimensional metrology instruments within NIST, and more than ten SRMs (473, 475, 1692, 1960, 1961, 1965, 2059, 2800, 5000, 5001), as well as other SRMs in development. However, even with top performance, the limiting uncertainty of the LSI must be reduced to fully meet the needs of NIST's most demanding customers for dimensional calibrations and SRMs — and near-term requirements for the semiconductor industry are even more stringent.

Project Objective:

The objectives of this project are twofold: to ensure continued operation of the facility (which is more that 40 years old) with reduced uncertainty, and to procure a new instrument that substantially decreases the dominant uncertainty due to index of refraction.

The NIST Linescale Interferometer is one of the leading facilities in the world providing realization and dissemination of the meter for ruler-like standards. Of the nearly forty national measurement labs that have participated in the two major key comparisons of the last decade, only the LSI and two others have completed both with all of their measurements retained in the final reference values (based on preliminary results). In addition, in the most recent key comparison the LSI had the lowest stated uncertainty of all labs by a factor of nearly two.

The performance and accuracy of the LSI has been validated through international comparisons, most recently the "NANO 3: Line Scale Standards" comparison (2003), performed under the auspices of the International Bureau of Weights and Measures (BIPM), and the EuroMet.L-K7 comparision (2010).

NanoMet work is focused on further increasing the reliability of the system, and reducing

Figure 1. Meter bars with traditional x-shaped cross section to confine measurement to neutral plane.

measurement uncertainties. We have also developed a proposal for a new instrument to replace the existing facility and substantially decrease the measurement uncertainty. Completing this procurement, which is complex, lengthy and subject to unique controls and scheduling requirements is the other principal objective of this project.

Technical approach:

The LSI has benefited from a long program of continuous improvement which has allowed it to function at a high level over decades for measurements of artifacts ranging from meter bars to grid plates (figure 1). One validation of the instrument's performance is participation in international key comparisons, the two most recent being the Nano3 comparison (2003), and the Euromet.L-K7.

In 2003 the WGDM-7 preliminary comparison on nanometrology (Nano3) was completed with the publication of the final report by the pilot lab, PTB. Eleven laboratories' measurements of two 300 mm scales were compared. The scales were made of Zerodur and fused silica, but were otherwise identical (Figure 2). The results showed the LSI's stated uncertainty to be among the very lowest, and fully consistent with the measured results (Figure 3).

Starting in 2006, Euromet (the regional metrology organization for Europe) began conducting in collaboration

"We use this mask as our golden standard so it gets sent continuously to all our site[s] to confirm that we measure to the same grid on our Leica/ Vistec IPRO 1,2 or 3 or Nikon 5i, 6i tools."

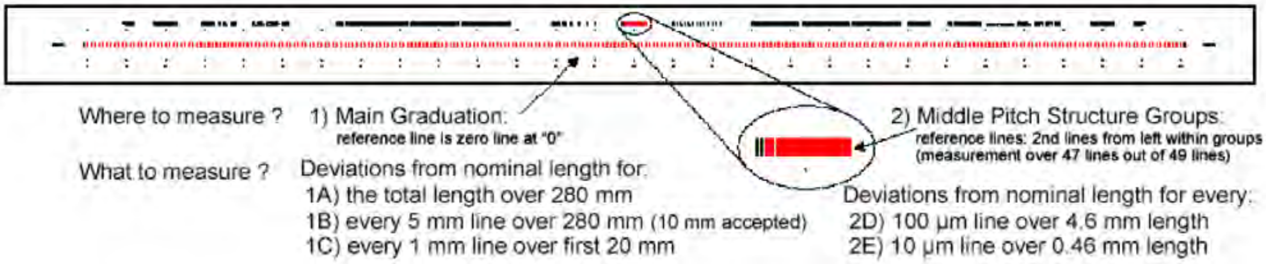

Figure 2. Nano 3 scale design.

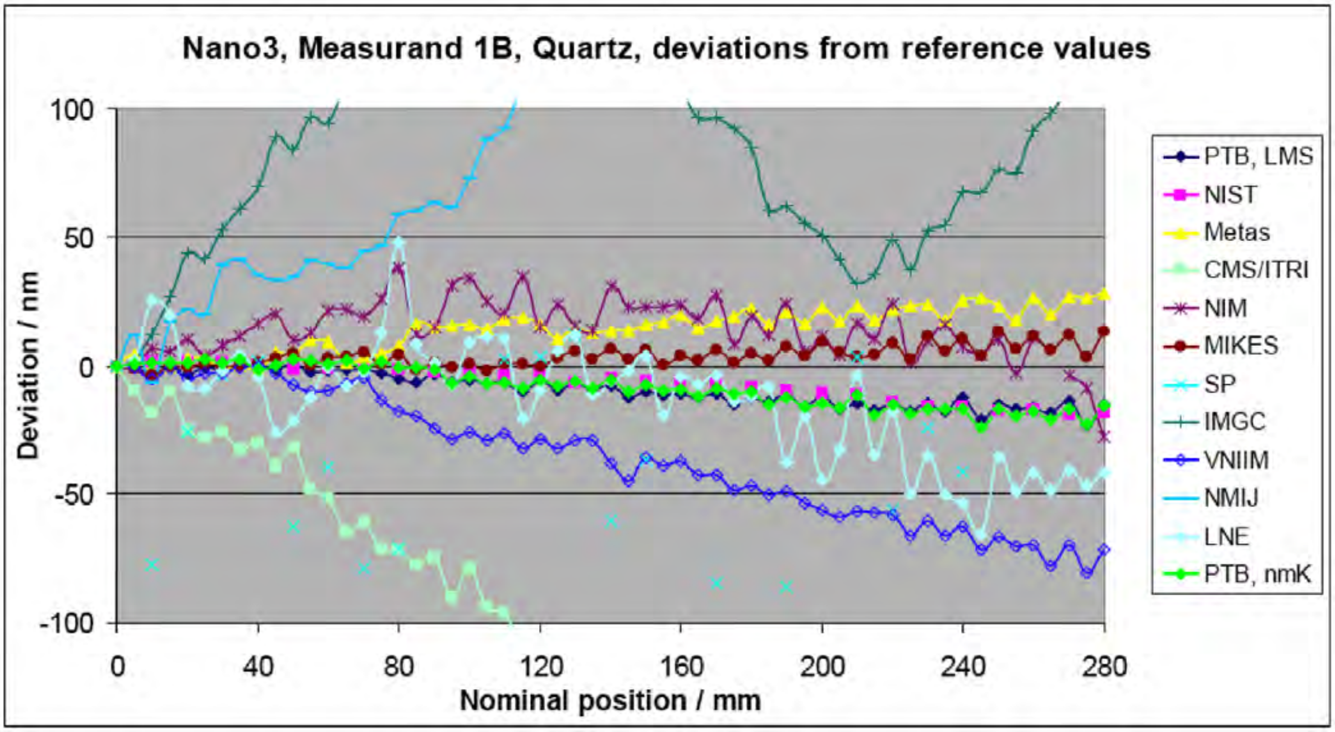

Figure 3. Example of Nano3 measurement results: 280 mm quartz scale, 5 mm increments. Deviation from weighted mean after excluding 3 participating labs.

with the CIPM a key comparison for length measurements on a 100 mm scale [fig. 4]. The Euromet.L-K7 comparison between 31 labs was organized by MIRS in Slovenia, and the final report is expected by 2010. Preliminary results show the LSI's measurements to have very low uncertainty and to be among the closest to the nominal reference values.

During the 5 years covered by this report, the LSI performed 74 calibrations, some of which have very high impact (see below). A single LSI calibration can directly control several hundred million dollars of industrial production around the world, and indirectly control billions of dollars of industrial production. This level of performance is particularly notable in light of the age of the instrument and the unavoidable failure and replacement of components that have long outlived their expected service lives. Upgrading a system in daily use that also provides a critical capability must follow the principle of "First do no harm". Subsystems or components most likely to require replacement are identified and new systems are designed to be tested and run in parallel with the existing components. The new systems are also designed to enhance the LSI performance. The targeted subsystems are: the line detection electronics, scale positioning servo control electronics, temperature measurement sensor array and electronics, temperature control electronics, and the fluid cooling, heating and circulation system. The line detection improvements have also been designed to improve the sensitivity for detecting sub-micrometer lines which are increasingly required. The upgraded temperature measurement and control system should also improve the temperature stability of the system, and decrease the uncertainty for long artifacts.

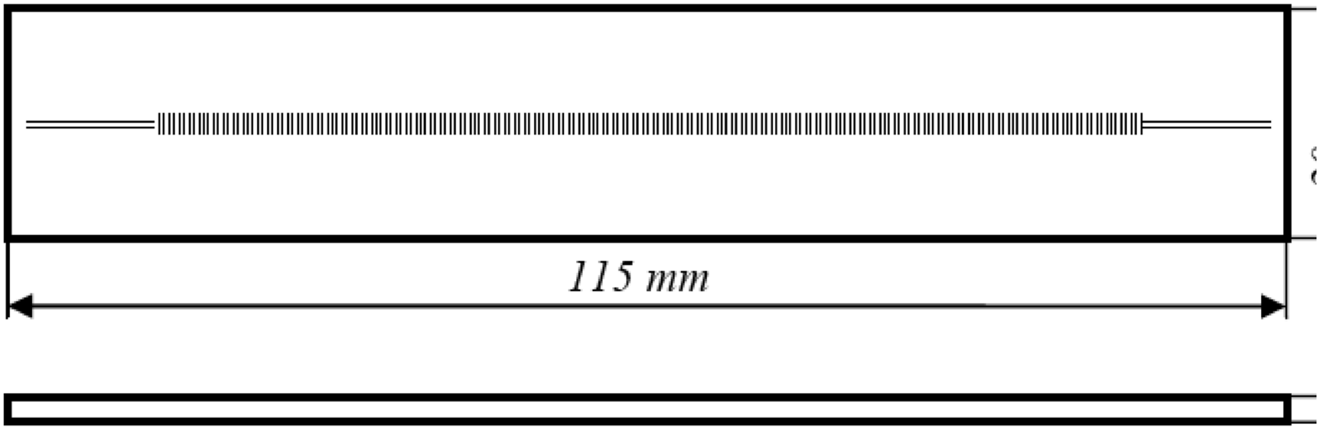

Figure 4. Euromet.L-K7 artifact

Impact/Benefit:

- "We use this mask as our golden standard so it gets sent continuously to all our site[s] to confirm that we measure to the same grid on our Leica/Vistec IPRO 1,2 or 3 or Nikon 5i, 6i tools." Dupont/Toppan Photomask — the worlds largest photomask supplier.

- As the top of the measurement chain for scale (ruler-like) measurements in the US, the impact of the LSI is significant. As well as providing traceability to the SI for more than ten SRMs that are critical to industries like Semiconductor manufacturing, a single calibration can have tremendous impact as demonstrated by the customer quote above

Accomplishments:

- The LSI was one of top performing instruments in the Nano3 key comparison.
- The LSI is one of the top instruments in the preliminary results of the Euromet.L-K7 key comparison.
- 74 calibrations performed — some with very high impact.
- World-class performance maintained despite periodic failures due to age.
- Successfully developed proposal to procure new LSI. Procurement currently in progress.

Publications:

- John S. Beers and William B. Penzes "The NIST Length Scale Interferometer", Journal of Research of the National Institute of Standards and Technology, Volume 3, May-June 1999, pp 225-252.

Presentations:

- T. LeBrun, "Design Considerations for a New NIST Linescale Interferometer", NIST, April 2009

Reports:

- William B. Penzes and Michael T. Postek, final report "Key Comparison of Nano-3 280 mm Line Scale, April, 2001.
- William B. Penzes, Thomas W. LeBrun and John A. Kramar, final report "EUROMET Key Comparison, EUROMET.L-K7 100 mm quartz Line Scale", March, 2008.

Measurement Reports:

- Seventy four measurement reports were issued in the Period FY04 – FY09

Customers:

- VLSI Standards,
- Nikon
- Northrop Grumman Space
- Pratt & Whitney
- Lockheed Martin
- Corning
- US DOE
- Mitutoyo America
- ALCOA
- US Customs Lab
- IBM

- L.S. Starret
- EuroMet
- Dupont/Toppan Photomask

Collaborators:

- Intel
- Nikon
- Los Alamos
- NASA
- Boeing
- University of North Carolina at Charlotte (New LSI development collaborator)

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Nanometer Scale Dimensional Metrology: Calibrated Atomic Force Microscope

Industry Need:

The semiconductor and nanotechnology industries have rapidly increasing dimensional metrology requirements in regimes where traceability to the SI unit of length is not always readily available. For example, the International Technology Roadmap for Semiconductors (ITRS) identifies dimensional metrology as a key enabling technology for the development of next-generation integrated circuits.

According to the 2007 update, the goal in 2009 for critical dimension (CD) measurement uncertainty for isolated lines was ± 0.42 nm; this demand tightens to ± 0.29 nm by 2012. Although most in-line metrology is performed using scanning electron microscopes (SEM) and scatterometry, these instruments are not presently capable of first-principles accuracy. That is, they must be calibrated using reference measurements from a tool or combination of tools which is capable of intrinsic accuracy. Such a tool is now referred to as a reference measurement system (RMS), and the 2007 update of the ITRS highlights the growing importance of an RMS. The use of atomic force microscope (AFM) and transmission electron microscope (TEM) cross measurements section for this purpose – often in combination – is now a fairly common practice in the industry.

To perform traceable dimensional metrology using commercially available instruments – such as atomic force microscopes or scanning electron microscopes – in the nanoscale regime requires a source of calibration such as a transfer standard. Although there are many commercial suppliers of such secondary standards, these suppliers rely on NIST to provide traceable calibration of their internal master standards.

The technical focus of this project, therefore, development and implementation of scanning probe microscope instrumentation for traceable dimensional metrology, is thus driven by the anticipated industry needs for reduced measurement uncertainty for in-line metrology tools such as the SEM and scatterometer – since these in turn rely on reduced measurement uncertainty for techniques such as AFM.

Project Objective:

The goal of this project is to meet the needs of users of scanned probe microscope (SPM) metrology in the nanotechnology and semiconductor industries – where relevant feature sizes continue to shrink and are now approaching atomic dimensions. In this regime, SPM metrology has been growing in importance and applications during the last two decades. To disseminate traceability in this regime, NIST has developed the calibrated atomic force microscope (C AFM) – an instrument with traceability to the SI meter in all three axes of motion. Dissemination of this traceability is achieved through reference measurements for internal and external customers and through participation in international measurement comparisons.

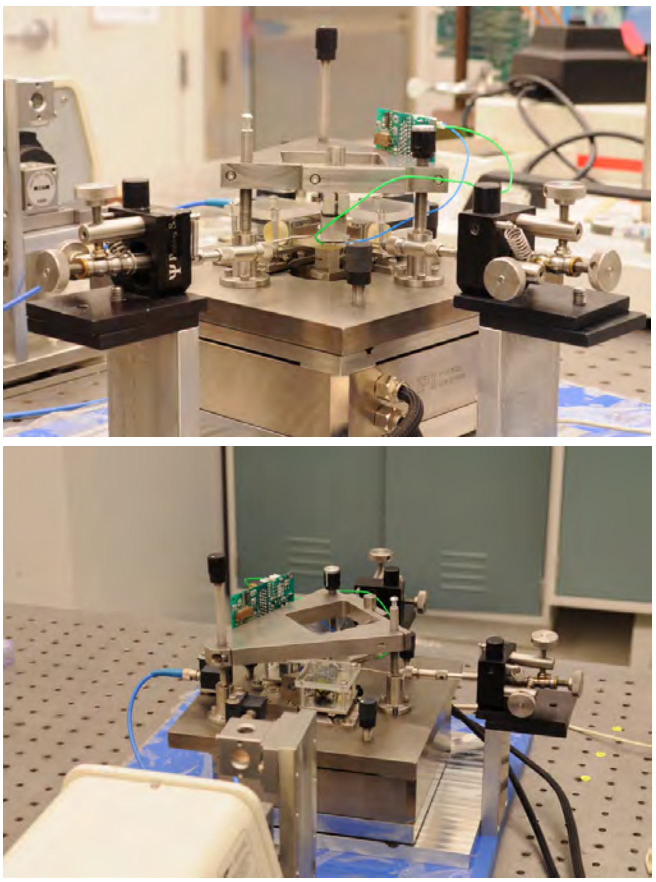

Figure 1. The NIST Calibrated Atomic Force Microscope.

Technical Approach:

The technical approach to the project goal, which is the development and implementation of scanning probe microscope instrumentation for traceable dimensional metrology, is to incorporate traceable interferometric displacement metrology with a custom, high accuracy

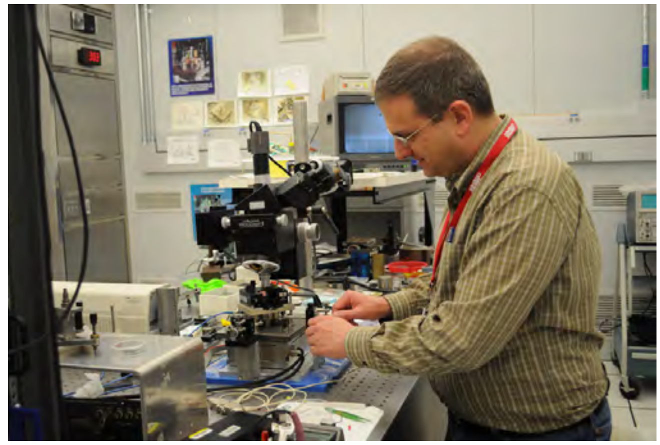

Figure 2. NIST staff scientist Ronald Dixson positions a sample for measurement in the C-AFM.

scanner – using a commercial AFM controller as a platform. Using this approach, NIST has developed the calibrated atomic force microscope (C AFM).

The C-AFM, now in its fourth generation, is a custom-built instrument that has built-in metrology on all three axes of motion traceable to the SI meter through the 633 nm wavelength of a He-Ne laser. It has been used to provide accurate pitch and step height metrology for customers internal and external to NIST, and we have participated

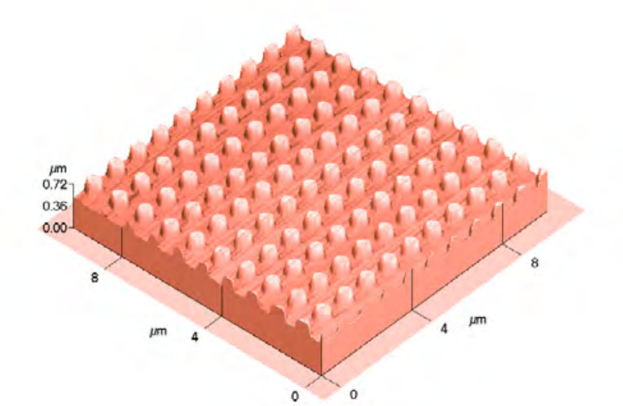

Figure 3. C-AFM image of a 1000 nm pitch two dimensional grating used in an international comparison.

in three international comparisons of dimensional nanometrology.

One international comparison involved step height. The other two involved one and two-dimensional grating period measurements. In the recent comparison of two-dimensional AFM pitch metrology, the C-AFM measurements had relative expanded uncertainty ($k = 2$) of about 1×10^{-3}.

.....the International Technology Roadmap for Semiconductors (ITRS) identifies dimensional metrology as a key enabling technology for the development of next-generation integrated circuits.

The recent release of NIST reference materials (RM) 8011, 8012, and 8013 was facilitated by C AFM reference measurements to support the higher throughput instruments used for the actual characterization. For the example, the SEM scale calibration was supported by a C-AFM measurement of a 100 nm pitch grating. The relative expanded uncertainty of the C-AFM measurement was 6×10^{-4}.

Impact/Benefit:

- Traceable nanoscale length measurements US nanotechnology industries directly or through commercial standards suppliers (e.g. VLSI Standards, K-TEK, Veeco Probes) or consortia (e.g. SEMATECH).
- Strategic collaborations with industrial users of SPM to improve traceability and accuracy of industrial manufacturing metrology.

Accomplishments:

- The C-AFM was used to perform a traceable 100 nm grating pitch measurement with 6×10^{-4} relative expanded uncertainty in support of SEM metrology of Au nano particles for NIST RM project.
- Commenced tri-lateral comparison of pitch measurements with A-Star (Singapore) and a commercial standards supplier (ASM).
- We completed NIST participation in an international round robin of two dimensional pitch measurements using the C-AFM. Two gratings – 1000 nm and 290 nm pitch – were measured.
- C-AFM step height measurements on an internal standard were used to provide a traceable calibration standard for use in MSEL during the Au nano particle project. The AFM height measurements on nanoparticle samples in MSEL were performed using this calibration.
- The C-AFM was used to support the CD-AFM reference measurement system (RMS) collaboration with SEMATECH. A 2 µm pitch grating measured on the C AFM was used for refinement of the scale calibration on the CD-AFM at SEMATECH.
- A step height measurement using the C-AFM was used to provide a z-axis reference value for collaborators at IBM – who used it for z-axis calibration of their CD-AFM.
- Evaluated a potential intrinsic SiC 1 nm height standard using the C-AFM.

Publication List:

2009

- Dixson, R., Fu, J., Orji, N., Renegar, T., Zheng, A., Vorburger, T., Hilton, A., Cangemi, M., Chen, L., Hernandez, M., Hajdaj, R., Bishop, M., Cordes, A., “Reference Metrology in a Research Fab: The NIST Clean Calibrations Thrust,” SPIE Proceedings Vol. 7272, 727209-1-12 (2009).
- Silver, R. M., Zhang, N. F., Barnes, B. M., Zhou, H., Heckert, A., Dixson, R., Germer, T. A., Bunday, B., “Improving Optical Measurement Accuracy using Multi-technique Nested Uncertainties,” SPIE Proceedings Vol. 7272, 727202-1-14 (2009).
- Attota, R., Stocker, M., Silver, R., Heckert, A., Zhou, H., Kasica, R., Chen, L., Dixson, R., Orji, N., Barnes, B., Limpscomb, P., “Through-focus Scanning and Scatterfield Optical Methods for Advanced Overlay Target Analysis,” SPIE Proceedings Vol. 7272, 727214-1-13 (2009).

2008

- Dixson, R., Potzick, J., Orji, N. “Recalibration of the SRM 2059 Master Standard using Traceable Atomic Force Microscope Metrology,” SPIE Proceedings Vol. 7122, 71222Q-1-12 (2008).
- Potzick, J., Dixson, R., Quintanilha, R., Stocker, M., Vladar, A., Buhr, E., Häßler-Grohne, W., Bodermann, B., Frase, C. G., Bosse, H., “International photomask linewidth comparison by NIST and PTB,” SPIE

Proceedings Vol. 7122, 71222P-1-14 (2008).

- Orji, N. G., Dixson, R. G., Bunday, B. D., Allgair, J. A., "Accuracy Considerations for Critical Dimension Semiconductor Metrology," SPIE Proceedings Vol. 7042, 70420A-1-11 (2008).
- Smith, S., Tsiamis, A., McCallum, M., Hourd, A. C., Stevenson, J. T. M., Walton, A. J., Dixson, R. G., Allen, R. A., Potzick, J. E., Cresswell, M. W., and Orji, N. G., "Comparison of Measurement Techniques for Advanced Photomask Metrology," Proceedings 2008 IEEE International Conference on Microelectronic Test Structures (ICMTS), pp. 35-39 (2008).
- Robert, J., Banke, W., Dixson, R., Strocchia-Rivera, C., "Traceable Calibration of AFM Step Height Measurements for Integrated Circuit Manufacturing," SPIE Proceedings Vol. 6922, 69223V-1-17 (2008).
- Orji, N. G., Dixson, R. G., Bunday, B. D., Allgair, J. A., "Towards Accurate Feature Shape Metrology," SPIE Proceedings Vol. 6922, 692208-1-9 (2008).
- Silver, R. M., Barnes, B., Heckert, A., Attota, R., Dixson, R. G., Jun, J., "Angle Resolved Optical Metrology," SPIE Proceedings Vol. 6922 , 6922M-1-12 (2008).
- Attota, R., Silver, R., Dixson, R., "Linewidth measurement technique using through-focus optical images," Applied Optics Vol. 47, 495-503 (2008).
- Patrick, H. J., Attota, R., Barnes, B., Germer, T. A., Dixson, R. G., Stocker, M. T., Silver, R. M., Bishop, M. R., "Optical critical dimension measurement of silicon grating targets using back focal plane scatterfield microscopy," J. Micro/Nanolith. MEMS MOEMS Vol. 7, 013012-1-11 (2008).
- Cresswell, M. W., Allen, R. A., Guthrie, W. F., Murabito, C. E., Dixson, R. G., Hunt, A., "Comparison of SEM and HRTEM CD Measurements Extracted from Test-Structures Having Feature Linewidths from 40 nm to 240 nm," IEEE Trans. Instrum. Meas. Vol. 57, 100-109 (2008).

2007

- Dixson, R., Orji, N. G., Potzick, J., Fu, J., Allen, R. A., Cresswell, M. W., Smith, S., Walton, A. J., Tsiamis, A., "Photomask Applications of Traceable Atomic Force Microscope Dimensional Metrology at NIST," SPIE Proceedings Vol. 6730, 67303D-1-13 (2007).
- Patrick, H. J., Germer, T. A., Cresswell, M. W., Allen, R. A., Dixson, R. G., and Bishop, M., "Modeling and Analysis of In-Plane and Out-of-Plane Target and Substrate Scatterometry Signatures for Optical Critical Dimension Reference Material Applications," Frontiers of Characterization and Metrology for Nanoelectronics:2007, AIP Conference Proceedings Vol. 931, 392 – 396 (2007).
- Allen, R. A., Cresswell, M. W., Dixson, R. G., Guthrie, W. F., Shulver, B. J. R., Stevenson, J. T. M., Walton, A. J., "CD Reference Materials Fabricated on Monolithic 200 mm Wafers for Automated Metrology Tool Applications," Frontiers of Characterization and Metrology for Nanoelectronics: 2007, AIP Conference Proceedings Vol. 931, 387 - 391 (2007).
- Chu, W., Fu, J., Dixson, R., Vorburger, T., "Linewidth Measurement Based on Automatically Matched and Stitched AFM Images," Frontiers of Characterization and Metrology for Nanoelectronics: 2007, AIP Conference Proceedings Vol. 931, 407-412 (2007).
- Orji, N. G., Bunday, B. D., Dixson, R. G., Allgair, J. A., "A Systematic Approach to Accurate Evaluation of CD-Metrology Tools," Proceedings 2007 IEEE International Conference on Microelectronic Test Structures (ICMTS), pp. 9-13 (2007).
- Allen, R. A., Patrick, H., Bishop, M., Germer, T. A., Dixson, R. G., Guthrie, W., Cresswell, M. W., "Study of Test Structures for Use as Reference Material in Optical Critical Dimension Applications," Proceedings 2007 IEEE International Conference on Microelectronic Test Structures (ICMTS), pp. 20-25 (2007).
- Dixson, R., Guthrie, W., Cresswell, M., Allen, R., Orji, N. G., "Single Crystal Critical Dimension Reference Materials (SCCDRM): Process Optimization for the Next Generation of Standards," SPIE Proceedings Vol. 6518, 651816-1-11 (2007).
- Dixson, R., Orji, N. G., "Comparison and Uncertainties of Standards for Critical Dimension Atomic Force Microscope Tip Width Calibration," SPIE Proceedings Vol. 6518, 651816-1-11 (2007).
- Robert, J., Banke, W., Dixson, R., "Leveraging LER to Minimize Linewidth Measurement Uncertainty in a Calibration Exercise," SPIE Proceedings Vol. 6518, 65184T-1-15 (2007).
- Orji, N. G., Dixson, R. G., Martinez, A., Bunday, B. D., Allgair, J. A., and Vorburger, T. V., "Progress on implementation of a reference measurement system based on a critical-dimension atomic force microscope," J. Micro/Nanolith. MEMS MOEMS Vol. 6, 023002-1-10 (2007).

- Orji, N. G., Dixson, R. G., "Higher order tip effects in traceable CD-AFM-based linewidth measurements," Meas. Sci. Technol. 18, 448-455 (2007).
- Orji, N. G., Dixson, R., Bunday, B. D., Allgair, J. A., "TEM Calibration Methods for Critical Dimension Standards," SPIE Proceedings Vol. 6518, 651810-1-10 (2007).
- Qian, X., Villarrubia, J., Tian, F., Dixson, R., "Image Simulation and Surface Reconstruction of Undercut Features in Atomic Force Microscopy," SPIE Proceedings Vol. 6518, 651811-1-12 (2007).
- Park, B. C., Choi, J., Ahn, S. J., Kim, D. H., Lyou, J., Dixson, R., Orji, N. G., Fu, J., Vorburger, T. V., "Applications of Carbon Nanotube Probes in a Critical Dimension Atomic Force Microscope," SPIE Proceedings Vol. 6518, 651819-1-14 (2007).

2006

- Dixson, R., Orji, N. G., Fu, J., Cresswell, M., Allen, R., Guthrie, W., "Traceable Atomic Force Microscope Dimensional Metrology at NIST," SPIE Proceedings Vol. 6152, 61520P-1-11 (2006).
- Cresswell, M., Guthrie, W., Dixson, R., Allen, R. A., Murabito, C. E., Martinez de Pinillos, J. V., "RM8111: Development of a Prototype Linewidth Standard," J. Res. Natl. Inst. Stand. Technol. 111, 187 – 203 (2006).
- Orji, N., Martinez, A., Dixson, R., Allgair, J., "Progress on Implementation of a CD-AFM based Reference Measurement System," SPIE Proceedings Vol. 6152, 61520O-1-12 (2006).
- Attota, R., Silver, R., Bishop, M., Dixson, R., "Optical Critical Dimension Measurement and Illumination Analysis Using the Through-focus Focus Metric," SPIE Proceedings Vol. 6152, 61520K-1-11 (2006).

2005

- Cresswell, M. W., Park, B., Allen, R. A., Guthrie, W. F., Dixson, R. G., Tan, W., Murabito, C. E., "Comparison of SEM and HRTEM CD-Measurements Extracted from Monocrystalline Test Structures Having Feature Linewidths from 40 nm to 240 nm," Proceedings 2005 IEEE International Conference on Microelectronic Test Structures (ICMTS), pp.6-12 (2005).
- Dixson, R., Fu, J., N. Orji, Guthrie, W., Allen, R., Cresswell, M., "CD-AFM Reference Metrology at NIST and SEMATECH", SPIE Proceedings Vol. 5752, 324-336 (2005).
- Dixson, R. G., Allen, R. A., Guthrie, W. F., and Cresswell, M. W., "Traceable Calibration of Critical-Dimension Atomic Force Microscope Linewidth Measurements with Nanometer Uncertainty" J. Vac. Sci. Technol. B Vol. 23, 3028-3032 (2005).
- Cresswell, M., Dixson, R., Guthrie, W., Allen, R., Murabito, C., Park, B., Martinez, J., Hunt, A., "Critical Dimension Reference Features with Sub-Five Nanometer Uncertainty," SPIE Proceedings Vol. 5752, 288-303 (2005).
- Orji, N. G., Vorburger, T. V., Fu, J., Dixson, R., Raja, J., Nguyen, C. V., "Line Edge Roughness Metrology using Atomic Force Microscopes", Meas. Sci. Technol. 16, 2147-2154 (2005).

Presentations:

Panel Discussions

- Dixson, R., Organized and Chaired a Panel Discussion on "Contour Metrology" at the SPIE Advanced Lithography Conference, San Jose, CA, Feb. 26, 2009.
- Dixson, R., Organized and Co-Chaired a Panel Discussion on "Reference Metrology" at the SPIE Advanced Lithography Conference, San Jose, CA, Feb. 28, 2008.
- Dixson, R., member of panel for "Global Collaboration on Reference Metrology," Scanning Conference, Gaithersburg, MD, April 16, 2008.
- Dixson, R., Organized and Chaired an evening panel session on "CD Standards: Past, Present, and Future" at the SPIE Advanced Lithography Conference, San Jose, CA, March 1, 2007

Invited

- Dixson, R., "Traceable AFM Dimensional Metrology at NIST," NIST Workshop on Calibrations and Standards For Nanomechanical Measurements, Gaithersburg, MD, June 16, 2009.
- Dixson, R., "Reference Metrology in a Research Fab: The NIST Clean Calibrations Program,"SEMATECH Advanced Metrology Advisory Group, Austin, TX, September 24, 2008.
- Dixson, R., "NIST Single Crystal Critical Dimension Reference Materials: The Next Generation and the NanoCD Connection," SEMATECH Advanced Metrology Advisory Group, Austin, TX, September 19, 2006.
- Dixson, R., "NIST Single Crystal Critical Dimension Reference Materials: Retrospective on the 2004

Release," SEMATECH Advanced Metrology Advisory Group, Napa, CA, February 16, 2006.

- Dixson, R., "CD-AFM Reference Measurement System," SEMATECH Advanced Metrology Advisory Group, Austin, TX, February 22, 2005.

Contributed

- Dixson, R., "Reference Metrology in a Research Fab: The NIST Clean Calibrations Thrust," SPIE Advanced Lithography Conference, San Jose, CA, Feb. 23, 2009.
- Dixson, R., "Recalibration of the SRM 2059 Master Standard using Traceable Atomic Force Microscope Metrology," SPIE Photomask Technology Conference, Monterey, CA, October 9, 2008.
- Dixson, R., "Photomask Applications of Traceable Atomic Force Microscope Dimensional at NIST," SPIE Photomask Technology Conference, Monterey, CA, September 20, 2007.
- Dixson, R., "Single Crystal Critical Dimension Reference Materials (SCCDRM): Process Optimization for the Next Generation of Standards," SPIE Advanced Lithography Conference, San Jose, Feb. 28, 2007.
- Ronald Dixson, "Comparison and Uncertainties of Standards for Critical Dimension Atomic Force Microscope Tip Width Calibration," SPIE Advanced Lithography Conference, San Jose, CA, Feb. 28, 2007.
- Dixson, R., "Traceable Atomic Force Microscope Dimensional Metrology at NIST," SPIE Microlithography Conference, San Jose, CA, Feb. 21, 2006.
- Dixson, R., "CD-AFM Reference Metrology at NIST and SEMATECH", SPIE Microlithography Conference, San Jose, CA, March 1, 2005.
- Dixson, R., "Traceable Calibration of Critical-Dimension Atomic Force Microscope Linewidth Measurements with Nanometer Uncertainty," 49th International Conference on Electron, Ion, Photon Beam and Nanotechnology, Orlando, FL, June 2, 2005.

Awards:

- Bronze medal for development of the Reference Measurement System, a probe microscope at SEMATECH, providing dimensional calibrations for the semiconductor industry.

Customers:

- SEMATECH/ISMI
- VLSI Standards
- Veeco Probes (Nanodevices)
- K-TEK

Collaborators:

- SEMATECH/ISMI
- PTB
- VLSI Standards
- Advanced Surface Microscopy
- NRC-INMS

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ron.dixson@nist.gov

Wafer Level AFM Metrology for Critical Dimension Measurements.

Industry Need:

"An RMS is well characterized using the best science and technology of dimensional metrology can offer: applied physics, sound statistics, and proper handling of all measurement error contributions. …Through its measurements this "golden" instrument can help production and reduce costs across the company or companies." International Technology Roadmap for Semiconductors, Metrology Section, p. 38 (2007).

"Calibration of inline CD metrology equipment requires careful implementation of the calibration measurement equipment referred to as reference metrology. For example, laboratory based TEM or CD-AFM must have precision that matches or exceeds inline CD and have to be frequently calibrated." International Technology Roadmap for Semiconductors, Metrology Section, p. 11 (2007).

Traceable nanoscale length metrology for semiconductor fabrication facilities.

Procedures, samples and methods development for fundamental length relate measurement problems.

To address traceability problems in semiconductor dimensional metrology, the National Institute of Standards and Technology (NIST) in collaboration with SEMATECH developed and implemented a critical-dimension atomic force microscope (CDAFM)-based reference measurement system (RMS). The key goal of the system is to transfer traceable length measurements to tools used in the semiconductor industry.

Traditionally, the semiconductor industry placed a greater emphasis on precision and tool matching than on accuracy. But as the size of features being measured continue to get smaller, and with recent results that linked important parameters such as line edge roughness to device performance, the importance of traceability cannot be overemphasized. In addition to traceability transfer, the RMS is also used when comparing the performance of different metrology tools at the same or different location, and when verifying the reliability of models such as optical proximity correction. The nature of the RMS requires that it have a higher level of performance and stability than the tools it supports. Our system has since supported numerous applications and continues to do. In addition to developing CD-AFM measurement and analysis methods for the industry, we have also advised and helped users implement CD-AFM systems.

Project Objective:

The goal of this project is to develop traceable CD-AFM based dimensional measurement systems for calibration of linewidth, step height, pitch, and related features, and disseminate the measurements and underlying technology to industry.

A key focus of this project is to transfer traceable methods developed by NIST personnel to semiconductor fabrication facilities. To do this, we developed a CD-AFM based reference measurement system, which is a dedicated instrument with low uncertainty that is used to transfer traceability to in-line metrology tools. This instrument was developed in collaboration with SEMATEC/ISMI. The NIST/ISMI CD-AFM-based RMS provides rigorously traceable measurements for linewidth, height, pitch, and sidewall angle. These measurements lend SI traceability to a wide range of production relevant samples. The instrument is calibrated using Si lattice spacing imaged by annular dark field transmission electron microscope (ADF-TEM), and samples measured using the NIST calibrated atomic force microscope with interferometry in all three axes. We have also started another project on nanoscale lateral calibration using Si(111) as a reference.

"…through its measurements this "golden" instrument can help production and reduce costs across the company or companies."

In addition to measurements, and procedures development, we also play a key role in disseminating SI length metrology techniques through organizing workshops, panel discussions, and teaching short courses to industry.

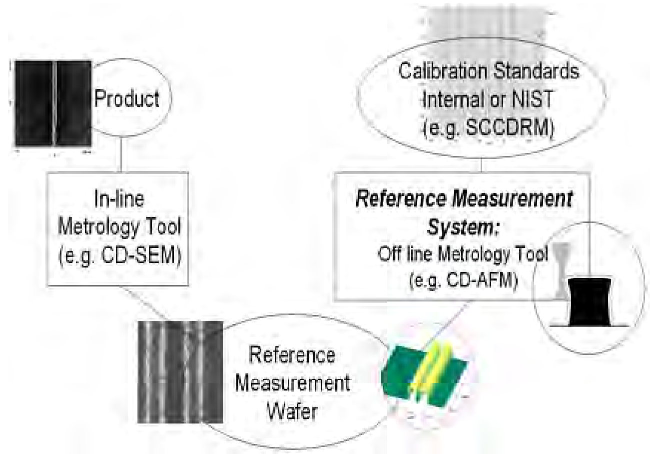

Figure 1. Based on a critical dimension atomic force microscope (CD-AFM) the reference measurement system is used to impart traceability to dimensional measurements made in semiconductor manufacturing facilities.

Technical approach:

- Introduce reference metrology techniques to production relevant measurements.
- Use methods such as ADF-TEM with aberration correction to introduce traceability to width measurements.
- Develop characterization and evaluation methods for key industry measurements such a sidewall angle, line edge roughness, and contact holes.
- Equipment Used: Critical dimension atomic force microscope; Annular dark field Transmission electron microscope (Collaborator)

Impact/Benefit:

- Introduction and dissemination of SI traceable width samples to SEMATECH member companies.
- Development and dissemination of SI traceable methods for CD-AFM-based sidewall angle measurements.
- Disseminated methods for implementing a reference measurement system to US semiconductor industry.
- Strategic collaboration with industry on development projects that leverage NIST expertise and introduce traceability to key measurements.

Figure 2. NIST CD AFM instrument. based reference measurement system within the Advanced Measurement Laboratory. One of the instruments used to support the dimensional metrology needs of the semiconductor industry.

Accomplishments:

- Developed of a reference measurement system at SEMATECH.
- Developed a rigorously traceable linewidth calibration system.
- Developed a rigorously traceable sidewall angle calibration method. First publication of procedures and methods for traceable characterization of nanoscale sidewall angle. (FY08, 09)
- Completed extensive CD-AFM and ADF-HRTEM measurements for TEM/CD_AFM comparison project. This work was done in collaboration with SEMATECH and Oakridge National Lab, and further reduces the uncertainty of our linewidth measurements. (FY08,09)
- Developed measurement procedures and requirements to characterize the stability and performance of carbon nanotube tips when used on automated atomic force microscopes. We are currently providing support to companies through SEMATECH. (FY08, 09)
- SEMATECH Benchmarking measurement support. We continued measurement and development support for SEMATECH metrology program by providing reference measurements for litho metrology projects. These projects include EUV resist evaluation, iArF photoresist SEM characterization, OCD/Scatterometry benchmarking and evaluation. Currently advising SEMATECH on these types of measurements.

- Provided CD-AFM measurement procedures development and support to SEMATECH member companies in the areas of CD metrology, overlay, mask metrology, resist imaging, MEMS imaging, finFETS, and nanoimprint features characterization.
- Completed measurements and reduced the uncertainties for photomask length metrology, and published the results.
- Completed measurements for NMI line width comparison.
- Evaluated and characterized a new SEMATECH CD-AFM using NIST developed methodologies. The instrument is currently operational in Albany New York is being used to perform measurements for member companies.
- Evaluated the use of a potential intrinsic SiC 1 nm height standard in a semiconductor environment using an automated instrument. We delivered a short report to the sample manufacturer – NASA .
- Developed methods to estimate some of the uncertainty due to higher order tip effects in critical dimension atomic force microscopes (CD-AFM) metrology. (FY04 to FY09)

Publication List:

2009

- Orji N. G, Ronald G. Dixson, Aaron Cordes, Benjamin D. Bunday, and John A. Allgair "Measurement traceability and quality assurance in a nanomanufacturing environment"Proc. SPIE, Vol. 7405, 740505 (2009);
- Dixson R., J. Fu, N. Orji, T. Renegar, A. Zheng, T. Vorburger, A. Hilton, M. Cangemi, L. Chen, M. Hernandez, R. Hajdaj, M. Bishop, A. Cordes, "Reference Metrology in a Research Fab: The NIST Clean Calibrations Thrust," SPIE Proceedings Vol. 7272, 727209-1-12 (2009).
- Attota R., M. Stocker, R. Silver, A. Heckert, H. Zhou, R. Kasica, L. Chen, R. Dixson, G. Orji, B. Barnes, P. Limpscomb, "Through-focus Scanning and Scatterfield Optical Methods for Advanced Overlay Target Analysis," SPIE Proceedings Vol. 7272, 727214-1-13 (2009).

2008

- Orji N. G., R. G. Dixson, B. D. Bunday, J. A. Allgair, "Accuracy Considerations for Critical Dimension Semiconductor Metrology," SPIE Proceedings Vol. 7042, 70420A-1-11 (2008).
- N. G. Orji, R. G. Dixson, B. D. Bunday, J. A. Allgair, "Towards Accurate Feature Shape Metrology," SPIE Proceedings Vol. 6922, 692208-1-9 (2008).
- Bunday B., A. Cordes, N. G. Orji, E.Piscani, D. Cochran, J. Byers, J. Allgair, B. J. Rice, Y. Avitan, R. Peltinov; M. Bar-zvi; O. Adan "Characterization of CD-SEM metrology for iArF photoresist materials" Proc. SPIE Vol. 6922 (2008)
- Dixson R., J. Potzick, N. Orji, "Recalibration of the SRM 2059 Master Standard using Traceable Atomic Force Microscope Metrology," SPIE Proceedings Vol. 7122, 71222Q-1-12 (2008).
- Potzick J., R. Dixson, R. Quintanilha, M. Stocker, A. Vladar, E. Buhr, W. Häßler-Grohne, B. Bodermann, C. G. Frase, H. Bosse, "International photomask linewidth comparison by NIST and PTB," SPIE Proceedings Vol. 7122, 71222P-1-14 (2008).
- Smith S., A.Tsiamis, M. McCallum, A.C. Hourd, J.T.M. Stevenson, A.J. Walton, R.G. Dixson, R.A. Allen, J.E. Potzick, M.W. Cresswell and N.G. Orji, "Comparison of Measurement Techniques for Advanced Photomask Metrology," Proceedings 2008 IEEE International Conference on Microelectronic Test Structures (ICMTS), pp. 35-39 (2008).
- Robert J, Bill Banke, Ronald Dixson, Carlos Strocchia-Rivera, "Traceable Calibration of AFM Step Height Measurements for Integrated Circuit Manufacturing," SPIE Proceedings Vol. 6922, 69223V-1-17 (2008).
- Silver R. M., B. Barnes, A. Heckert, R. Attota, R. G. Dixson, J. Jun, "Angle Resolved Optical Metrology," SPIE Proceedings Vol. 6922 , 6922M-1-12 (2008).
- Attota R., R. Silver, R. Dixson, "Linewidth measurement technique using through-focus optical images," Applied Optics Vol. 47, 495-503 (2008).
- Patrick H. J., R. Attota, B. Barnes, T. A. Germer, R. G. Dixson, M. T. Stocker, R. M. Silver, M. R. Bishop, "Optical critical dimension measurement of silicon grating targets using back focal plane scatterfield microscopy," J. Micro/Nanolith. MEMS MOEMS Vol. 7, 013012-1-11 (2008).
- Cresswell M. W., R. A. Allen, W. F. Guthrie, C. E. Murabito, R. G. Dixson, A. Hunt, "Comparison of SEM

and HRTEM CD Measurements Extracted from Test-Structures Having Feature Linewidths from 40 nm to 240 nm," IEEE Trans. Instrum. Meas. Vol. 57, 100-109 (2008).

- Clarke J. S., M. B. Schmidt, and N. G. Orji "Photoresist cross-sectioning with negligible damage using a dual-beam FIB-SEM: A high throughput method for profile imaging" J. Vac. Sci. Technol. B 25, p2526 Nov/Dec 2007

2007

- Orji N. G., R. G. Dixson, A. Martinez, B. D. Bunday, J. A. Allgair, and T. V. Vorburger, "Progress on implementation of a reference measurement system based on a critical-dimension atomic force microscope," J. Micro/Nanolith. MEMS MOEMS Vol. 6, 023002-1-10 (2007).
- Orji N. G., R. G. Dixson, "Higher order tip effects in traceable CD-AFM-based linewidth measurements," Meas. Sci. Technol. 18, 448-455 (2007).
- Orji N. G.,. R. Dixson, B. D. Bunday, J. A. Allgair, "TEM Calibration Methods for Critical Dimension Standards," SPIE Proceedings Vol. 6518, 651810-1-10 (2007).
- Orji N. G., B. D. Bunday, R. G. Dixson, J. A. Allgair, "A Systematic Approach to Accurate Evaluation of CD-Metrology Tools," Proceedings 2007 IEEE International Conference on Microelectronic Test Structures (ICMTS), pp. 9-13 (2007).
- Bunday B. , J. Allgair, E. Solecky, C. Archie, N. G. Orji, J. Beach, O. Adan, R. Peltinov, M. Bar-zvi, and J. Swyers "The coming of age of tilt CD-SEM" Proc. SPIE 6518, 65181S (2007)
- Dixson R., Ndubuisi G. Orji, James Potzick, Joseph Fu, Richard A. Allen, Michael Cresswell, Stewart Smith, Anthony J. Walton, Andreas Tsiamis, "Photomask Applications of Traceable Atomic Force Microscope Dimensional Metrology at NIST," SPIE Proceedings Vol. 6730, 67303D-1-13 (2007).
- Dixson R., W. Guthrie, M. Cresswell, R. Allen, N. G. Orji, "Single Crystal Critical Dimension Reference Materials (SCCDRM): Process Optimization for the Next Generation of Standards," SPIE Proceedings Vol. 6518, 651816-1-11 (2007).
- Dixson R., Ndubuisi G. Orji, "Comparison and Uncertainties of Standards for Critical Dimension Atomic Force Microscope Tip Width Calibration," SPIE Proceedings Vol. 6518, 651816-1-11 (2007).
- Patrick H. J., T. A. Germer, M. W. Cresswell, R. A. Allen, R. G. Dixson, and M. Bishop, "Modeling and Analysis of In-Plane and Out-of-Plane Target and Substrate Scatterometry Signatures for Optical Critical Dimension Reference Material Applications," Frontiers of Characterization and Metrology for Nanoelectronics:2007, AIP Conference Proceedings Vol. 931, 392 – 396 (2007).
- Allen R. A., M. W. Cresswell, R. G. Dixson, W. F. Guthrie, B. J. R. Shulver, J. T. M. Stevenson, A. J. Walton, "CD Reference Materials Fabricated on Monolithic 200 mm Wafers for Automated Metrology Tool Applications," Frontiers of Characterization and Metrology for Nanoelectronics: 2007, AIP Conference Proceedings Vol. 931, 387 - 391 (2007).
- Chu W., J. Fu, R. Dixson, T. Vorburger, "Linewidth Measurement Based on Automatically Matched and Stitched AFM Images," Frontiers of Characterization and Metrology for Nanoelectronics: 2007, AIP Conference Proceedings Vol. 931, 407-412 (2007).
- Allen R. A., H. Patrick, M. Bishop, T. Avery Dr. Germer, R. G. Dixson, W. Guthrie, M. W. Cresswell, "Study of Test Structures for Use as Reference Material in Optical Critical Dimension Applications," Proceedings 2007 IEEE International Conference on Microelectronic Test Structures (ICMTS), pp. 20-25 (2007).
- Robert J., Bill Banke, Ronald Dixson, "Leveraging LER to Minimize Linewidth Measurement Uncertainty in a Calibration Exercise," SPIE Proceedings Vol. 6518, 65184T-1-15 (2007).
- Qian X., J. Villarrubia, F. Tian, R. Dixson, "Image Simulation and Surface Reconstruction of Undercut Features in Atomic Force Microscopy," SPIE Proceedings Vol. 6518, 651811-1-12 (2007).
- Park B. C., J. Choi, S. J. Ahn, D. H. Kim, J. Lyou, R. Dixson, N. G. Orji, J. Fu, T. V. Vorburger, "Applications of Carbon Nanotube Probes in a Critical Dimension Atomic Force Microscope," SPIE Proceedings Vol. 6518, 651819-1-14 (2007).

2006

- Allgair J. A., B. D. Bunday, M Bishop, P. Lipscomb, and N. G. Orji "Litho-metrology challenges for the 45-nm technology node and beyond" Proc. SPIE 6152, 61520C-1-15 (2006).
- Orji N. G., A. Martinez, R. Dixson, J. Allgair, "Progress on Implementation of a CD-AFM based Reference Measurement System," SPIE Proceedings Vol. 6152 , 61520O-1-12 (2006).

- Dixson R., N. G. Orji, J. Fu, M. Cresswell, R. Allen, W. Guthrie, "Traceable Atomic Force Microscope Dimensional Metrology at NIST," SPIE Proceedings Vol. 6152, 61520P-1-11 (2006).
- Cresswell M., W. Guthrie, R. Dixson, R. A. Allen, C. E. Murabito, J. V. Martinez de Pinillos, "RM8111: Development of a Prototype Linewidth Standard," J. Res. Natl. Inst. Stand. Technol. 111, 187 – 203 (2006).
- Attota R., R. Silver, M. Bishop, R. Dixson, "Optical Critical Dimension Measurement and Illumination Analysis Using the Through-focus Focus Metric," SPIE Proceedings Vol. 6152, 61520K-1-11 (2006).

2005

- Cresswell M. W., B. Park, R. A. Allen, W. F. Guthrie, R. G. Dixson, W. Tan, C. E. Murabito, "Comparison of SEM and HRTEM CD-Measurements Extracted from Monocrystalline Test Structures Having Feature Linewidths from 40 nm to 240 nm," Proceedings 2005 IEEE International Conference on Microelectronic Test Structures (ICMTS), pp.6-12 (2005).
- Dixson R., J. Fu, N. Orji, W. Guthrie, R. Allen, M. Cresswell, "CD-AFM Reference Metrology at NIST and SEMATECH", SPIE Proceedings Vol. 5752, 324-336 (2005).
- Dixson R.,, R. A. Allen, W. F. Guthrie, and M. W. Cresswell, "Traceable Calibration of Critical-Dimension Atomic Force Microscope Linewidth Measurements with Nanometer Uncertainty" J. Vac. Sci. Technol. B Vol. 23, 3028-3032 (2005).
- Cresswell M. W., R. Dixson, W. Guthrie, R. Allen, C. Murabito, B. Park, J. Martinez, A. Hunt, "Critical Dimension Reference Features with Sub-Five Nanometer Uncertainty," SPIE Proceedings Vol. 5752, 288-303 (2005).
- Orji N. G., T. V. Vorburger, J. Fu, R. Dixson, J. Raja, C. V. Nguyen, "Line Edge Roughness Metrology using Atomic Force Microscopes", Meas. Sci. Technol. 16, 2147-2154 (2005).

Presentations:

Short Course

- N. G. Orji and B. Bunday "Introductory Metrology Course" ½ day short course to attendees of International SAMETCH Manufacturing Initiative - Manufacturing Symposium October 21 2008
- N. G. Orji and B. Bunday "Introductory Metrology Course" ½ day short course to attendees of International SAMETCH Manufacturing Initiative - Manufacturing Symposium October 22nd 2007

Panel Discussion

- Organized and Co-Chaired a Panel Discussion on "Contour Metrology" at the SPIE Advance Lithography Conference 2009
- "Global Collaboration in Reference Metrology" at the SPIE Advance Lithography Conference 2009
- Organized and Co-Chaired a Panel Discussion on "Reference Metrology" at the SPIE Advance Lithography Conference 2008
- N. G. Orji, "CD-AFM Applications in Semiconductor Metrology" Panel discussion on Reference Metrology Scanning 2008 April 16th 2008

Invited

- N. G. Orji, Ronald G. Dixson, Aaron Cordes, Benjamin D. Bunday, and John A. Allgair "Measurement traceability and quality assurance in a nanomanufacturing environment" Proc. SPIE, Vol. 7405, 740505 (2009);
- N.G. Orji "NIST Traceable AFM Program" SEMATECH Advanced Metrology Advisory Group (AMAG) meeting, Albany, September 10th 2009.
- N.G. Orji - NIST Reference Metrology Program - Global Collaboration on Reference Metrology - SPIE Advance Lithography Conference 2009
- N.G. Orji "NIST Traceable CD-AFM Program" SEMATECH Advanced Metrology Advisory Group (AMAG) meeting, Monterey CA, February 2009.
- N. G. Orji, "Carbon Nanotubes as AFM tips" 2nd Annual Tri-National Workshop on Standards for Nanotechnology February 6th 2008
- N. G. Orji, "NIST-SEMATECH CD-AFM Reference Measurement System" SEMATECH AMAG February 20th 2008
- N. G. Orji, "Update on NIST –SEMATECH CD-AFM RMS Project" Program Advisory Group (PAG) Meetings. (February 23rd 2008)
- N. G. Orji, "NIST SEMATECHCDAFM Based Reference Measurement System" Scanning 2008 April 17th 2008
- N.G. Orji, "NIST-SEMATECH CD-AFM Reference

Measurement System" International Technology Roadmap for Semiconductors (ITRS) Metrology Technical Working Group meeting. San Francisco, CA. July 16th 2007

- N.G. Orji "Update on the CD-AFM Reference Measurement System" SEMATECH Advanced Metrology Advisory Group (AMAG) meeting, Austin TX, September 27th 2007.
- N.G. Orji, "NIST-SEMATECH CD-AFM Reference Measurement System" International Technology Roadmap for Semiconductors (ITRS) Metrology Technical Working Group meeting. San Francisco, CA. July 16th 2007
- N.G. Orji, "Current Projects with the CD-AFM Reference Measurement System" SEMATECH Metrology Program Advisory Group (PAG) meeting, Santa Clara, CA, March 2nd 2007.
- N.G. Orji, "Inter-method Comparisons of Reference Width" "NIST Workshop Critical Dimension Standards: The Past, Present, and Future. San Jose, CA March 1st 2007
- N.G. Orji "Update on the CD-AFM Reference Measurement System" SEMATECH Advanced Metrology Advisory Group (AMAG) meeting, Santa Cruz, CA. February 23rd 2007
- N.G. Orji, "Current Projects with the CD-AFM Reference Measurement System" SEMATECH Metrology Program Advisory Group (PAG) meeting, Austin TX, September 21st 2006.
- N.G. Orji, "Developments in CD-AFM Reference Measurement System" SEMATECH Advanced CD Metrology Advisory Group (AMAG) meeting, Austin TX September 19th 2006.
- N. G. Orji "Reference Measurement Systems: An Example." International Technology Roadmap for Semiconductors (ITRS) Metrology Technical Working Group meeting. San Francisco, CA. July 11th 2006
- N.G. Orji "CD-AFM Reference Measurement System – Status, Plans, Current Results" SEMATECH Metrology Program Advisory Group (PAG) meeting, Austin TX. June 22nd 2006.
- N.G. Orji "Status of the CD-AFM Reference Measurement System" SEMATECH Metrology Program Advisory Group (PAG) meeting, Austin TX March 9th 2006.
- N.G.Orji "Update on the CD-AFM Reference Measurement System" SEMATECH Advanced Metrology Advisory Group (AMAG) meeting, NAPA Valley, CA. February 16th 2006.

Contributed

- N. G. Orji, "Toward accurate feature shape metrology" SPIE Advanced Lithography Metrology, Inspection, and Process Control for Microlithography XXII Conference February 25th 2008.
- N. G. Orji, " Update on the Proposed Nano 6 Comparison" Working Group on Dimensional Metrology Discussion Group 7 – Nanometrology September 24th 2008
- N. G. Orji, "Collaboration between ISOI Technical Committee 201 SC 9 and ISO Technical Committee 213 WG16" Technical Committee 201 September 20th 2008
- N. G. Orji, "Collaboration between ISOI Technical Committee 201 SC 9 and ISO Technical Committee 213 WG16" Technical Committee 201 Sub Committee-9 Scanning Probe Microscopy Sept. 17th 2008
- N.G. Orji "NIST –SEMATECH CD-AFM RMS Project" Precision Engineering Division/NIST Gaithersburg MD, June 26th 2007.
- N. G. Orji, "A Systematic Approach to Accurate Evaluation of CD-Metrology Tools", IEEE -International Conference on Microelectronic Test Structure. March 20th 2007.
- N. G. Orji "TEM Calibration Methods for Critical Dimension Standards". SPIE Advanced Lithography. San Jose, CA February 28th 2007
- N.G. Orji "Proposals for Comparisons in Nanometrology – Silicon Single Atom Step Height Measurements and AFM based SCCDRM Linewidth Measurements" CCL – WGDM -7 Meeting Wabern, Switzerland. April 26th 2006
- N.G. Orji "Status of Linewidth Comparison—Nano1" CCL – WGDM -7 Meeting Wabern, Switzerland. April 26th 2006
- N.G. Orji "Higher Order Tip Effects in CD-AFM based Linewidth Measurement" Nanoscale 2006: Conference on Quantitative Microscopy and Nanoscale Calibration Standards and Methods. Wabern, Switzerland. April 24th 2006
- N.G. Orji, "Progress on Implementation of a CD-AFM-Based Reference Measurement System" SPIE Microlithography Symposium. San Jose, CA. February 21st 2006.

- James S. Clarke, Mike Schmidt and George Orji "Enabling Fast Cross-Sectioning of Photoresist with an in-fab FIB-SEM by using a CD-AFM to Calibrate Profiles" Intel Components Research Workshop September 2007.
- R. Dixson, J. Fu, N. Orji, T. Renegar, A. Zheng, T. Vorburger, A. Hilton, M. Cangemi, L. Chen, M. Hernandez, R. Hajdaj, M. Bishop, A. Cordes, "Reference Metrology in a Research Fab: The NIST Clean Calibrations Thrust," SPIE Proceedings Vol. 7272, 727209-1-12 (2009).
- R. Dixson, J. Potzick, N. Orji, "Recalibration of the SRM 2059 Master Standard using Traceable Atomic Force Microscope Metrology," SPIE Proceedings Vol. 7122, 71222Q-1-12 (2008).
- Ronald Dixson, Ndubuisi G. Orji, James Potzick, Joseph Fu, Richard A. Allen, Michael Cresswell, Stewart Smith, Anthony J. Walton, Andreas Tsiamis, "Photomask Applications of Traceable Atomic Force Microscope Dimensional Metrology at NIST," SPIE Proceedings Vol. 6730, 67303D-1-13 (2007).
- R. Dixson, W. Guthrie, M. Cresswell, R. Allen, N. G. Orji, "Single Crystal Critical Dimension Reference Materials (SCCDRM): Process Optimization for the Next Generation of Standards," SPIE Proceedings Vol. 6518, 651816-1-11 (2007).
- Ronald Dixson, Ndubuisi G. Orji, "Comparison and Uncertainties of Standards for Critical Dimension Atomic Force Microscope Tip Width Calibration," SPIE Proceedings Vol. 6518, 651816-1-11 (2007).
- Robert J., Bill Banke, Ronald Dixson, "Leveraging LER to Minimize Linewidth Measurement Uncertainty in a Calibration Exercise," SPIE Proceedings Vol. 6518, 65184T-1-15 (2007).
- R. Dixson, N. G. Orji, J. Fu, M. Cresswell, R. Allen, W. Guthrie, "Traceable Atomic Force Microscope Dimensional Metrology at NIST," SPIE Proceedings Vol. 6152, 61520P-1-11 (2006).
- R. Dixson, J. Fu, N. Orji, W. Guthrie, R. Allen, M. Cresswell, "CD-AFM Reference Metrology at NIST and SEMATECH", SPIE Proceedings Vol. 5752, 324-336 (2005).
- R. G. Dixson, R. A. Allen, W. F. Guthrie, and M. W. Cresswell, "Traceable Calibration of Critical-Dimension Atomic Force Microscope Linewidth Measurements with Nanometer Uncertainty" J. Vac. Sci. Technol. B Vol. 23, 3028-3032 (2005).

Awards:

Bronze medal for development of the Reference Measurement System, a probe microscope at SEMATECH, providing dimensional calibrations for the semiconductor industry.

Customers:

- SEMATECH/ISMI
- VLSI Standards
- Intel
- HP
- IBM

Collaborators:

- SEMATECH
- PTB
- IBM
- Veeco Instruments
- VLSI Standards

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Wafer-Level SEM Metrology for Critical Dimension Measurements: SEM Metrology

Industry Need:

"Scanning Electron Microscopy (SEM) – continues to provide at-line and in-line imaging … and CD measurements. Improvements are needed … at or beyond the 45* nm *generation … Determination of the real 3-D shape…will require continuing advances in existing microscopy …" International Technology Roadmap for Semiconductors, 2007

According to the 2008 data available from the Semiconductor Industry Association, the semiconductor electronics industry had worldwide sales of $248.6 billion. In the production of integrated circuits, the whole fabrication process hinges on the features' widths as fundamental dimensional characteristics. It was calculated a few years ago that one nanometer improvement in the production translates to one billion dollar made by faster and more chips.

The semiconductor industry needs SEM high-performance dimensional metrology methods and standard artifacts, specifically those related to instrument magnification calibration, performance and the measurement of linewidth, and feature size and shape. This entails a multidimensional program including: detailed research of the signal generation in the SEM, electron beam-sample interaction modeling, developing NIST metrology instruments for the certification of standards, and developing the necessary artifacts and calibration procedures. The manufacturing of present-day integrated circuits requires that certain measurements be made of structures with dimensions of 65 nm or less with a very high degree of precision. The accuracy of these measurements is also important, but more so in the development and pilot lines. The measurements of the minimum feature size, known as CD, are made to ensure proper device operation. The U.S. industry needs high-precision, accurate, shape-sensitive dimension measurement methods and relevant calibration standards. The SEM Metrology Project supports all aspects of this need since scanning electron microscopy is key microscopic technique used for this sub-100 nm metrology.

NIST, in collaboration with SEMATECH, is developing two Reference SEMs, which are among the most advanced and well-characterized instruments. The new wafer Reference SEM has been successfully established. Detailed and thorough work of combined measurements and optimization of the SEMs has been carried out and improvements are underway, which include the assessment of resolution, e-beam and signal transfer characteristics, distortion and noise characteristics in various working modes. These commercially available SEMs with significant investment in money, knowledge and extensive developmental work, were turned into outstanding dimensional metrology instruments. With these traceable and accurate top-down and cross sectional imaging and measurements can be carried out for the determination of three-dimensional shape and size of various structures, including those on full-size wafers masks and multiple other samples. Traceability is provided through laser interferometry and accuracy will be achieved by using world-class Monte Carlo modeling of SEM images of all measured structures. Full measurement uncertainty has been worked out, which can help the development and better use of all SEMs. The new high-performance imaging and measurement methods developed throughout this work are ready for implementation on other SEMs. The work continues to improve the uncertainty of the measurements and to develop customized measurement methods for various IC technology samples that minimize measurement uncertainty.

Project Objective:

It is a goal of this project is to provide the microelectronics industry with highly accurate scanning electron microscope measurement and modeling methods for shape-sensitive measurements and relevant calibration standards with nanometer-level resolution. To carry out SEM metrology instrumentation development, including improvements in electron and ion optical system, detection, sample stage, and vacuum system is another objective. Furthermore, it is also a goal to conduct research and development of new metrology techniques using digital imaging and networked measurement tools solutions to key metrology issues confronting the semiconductor lithography industry.

Technical Approach:

The Scanning Electron Microscope Metrology Project, a multidimensional project, is being executed through several thrusts fully supported by the semiconductor industry.

SEM Magnification Calibration Artifacts: Essential to SEM dimensional metrology is the calibration of the scale or magnification of the instrument. Standard Reference Material (SRM) 2120 is an SEM magnification standard that will function at the low beam voltages used in the semiconductor industry and high beam voltages used in other forms of microscopy Conventional optical lithography provides a chance for large amount of good quality samples produced inexpensively. Because of the improvements of this technology, it is now possible to produce the finest lines with close to 100 nm width and with 200 nm pitch values. The largest pitch is 1500 µm. In order to make this artifact available (while the final certification details are being completed) the artifact will be released as Reference Material (RM) 8820 (Fig. 1). This Reference Material is primarily intended to be used for X and Y scale (or magnification) calibrations from less than 10 times magnifications to more than 100 000 times magnifications in SEMs. It was designed to provide good contrast at low and high electron landing energies (accelerating voltages). Beyond testing scale calibration, it can be used for non-linearity measurements, especially at lower than 10 000 times magnifications. It can also be used for any other type of microscope, e.g., optical and scanning probe microscopes. Most SEMs require a set of calibration structures of different sizes to cover the full range of possible magnifications. This standard is designed to meet that need. A unit of RM 8820 consists of a 20 mm × 20 mm lithographically patterned silicon chip.

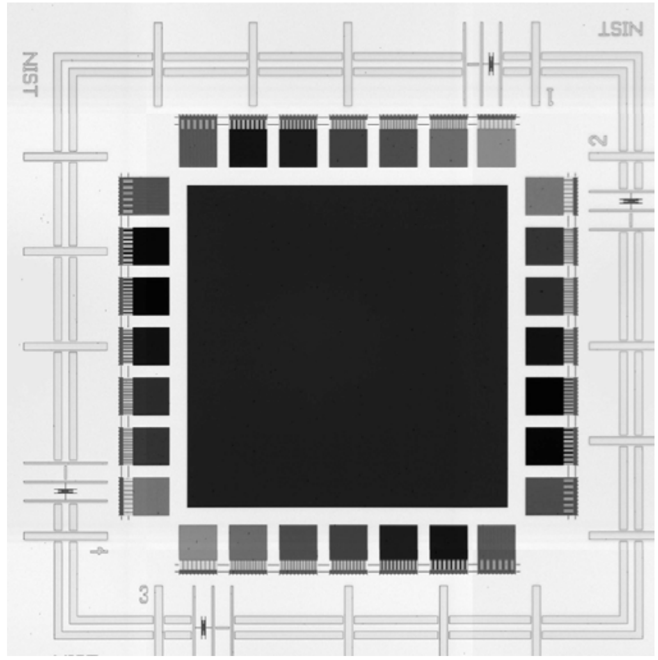

Figure 1. Optical microscope view of the 1.5 mm × 1.5 mm RM 8820 test structure.

SEM Performance Measurement Artifacts and Software Solutions: After comprehensive studies and experiments a plasma-etching Si called "grass" was chosen for Reference Material 8091. This sample has 5 nm to 25 nm size structures as is illustrated in Fig. 2. This effort included the development of the Reference Material 8091 and evaluation

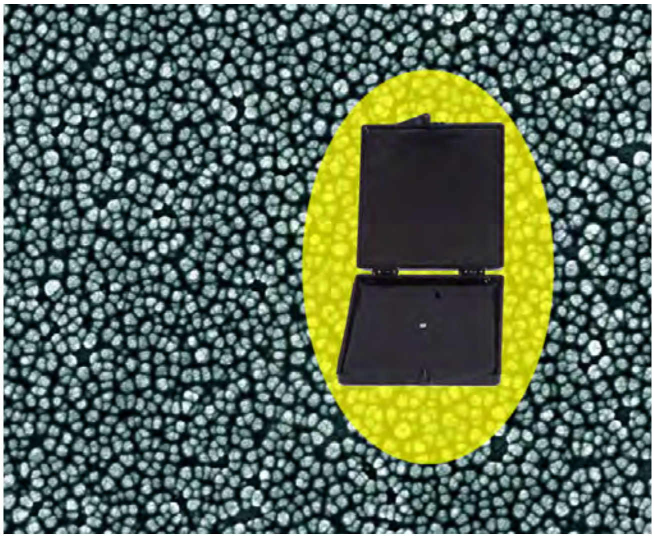

Figure 2. The RM 9081 Sharpness Reference Material

procedures suitable for correctly measuring image sharpness of scanning electron microscopes, especially of those that are used in the semiconductor industry. Currently we are working with ISO (International Standards Organization) to develop robust software solutions that will allow for resolution performance tracking of SEMs. The resolution performance characteristics of the SEM are particularly important to precise and accurate measurements needed for semiconductor processing. As a part of this effort the NIST SEM Resolution Measurement Reference Image Set was worked out to test the resolution measurement software themselves. This Reference Image Set contains a large number of artificial images that were made by taking onto account the amount and type of de-focusing, noise, vibration and drift and electron landing energies. Suitable samples are being sought to further improve this type of metrology (Fig. 3). Leading semiconductor-manufacturing companies has purchased several samples and these measurement artifacts are used in the benchmarking efforts of International SEMATECH. The existence of these samples fosters better understanding, objective measurement and enforcement of SEM spatial resolution performance.

SEM Linewidth Measurement Artifacts: Artifacts that are characterized and calibrated to the required small levels of uncertainty were and are in the focal point of the IC industry's dimensional metrology needs. Therefore, at NIST, it has been a longer-term goal to develop and deliver appropriate samples. For a long time, the possibilities were limited by the lack of various technologies available, especially the lack of accurate modeling methods. NIST, through several years of systematic efforts, developed Monte Carlo simulation-based modeling methods that

....commercially available SEMs with significant investment in money, knowledge and extensive developmental work, were turned into outstanding dimensional metrology instruments. With these traceable and accurate top-down and cross sectional imaging and measurements can be carried out for the determination of three-dimensional shape and size of various structures, including those on full-size wafers masks and multiple other samples. Traceability is provided through laser interferometry and accuracy will be achieved by using world-class Monte Carlo modeling of SEM images of all measured structures.

can deliver excellent results. These new methods can deduce the shape of integrated circuit structures from top-down view images through modeling and library-based measurement techniques with a few nm of accuracy. In several publications, NIST demonstrated the possibilities and described power of this measuring approach. Based on the newest results, now it is becoming possible to start to develop the long-awaited relevant line width standard for

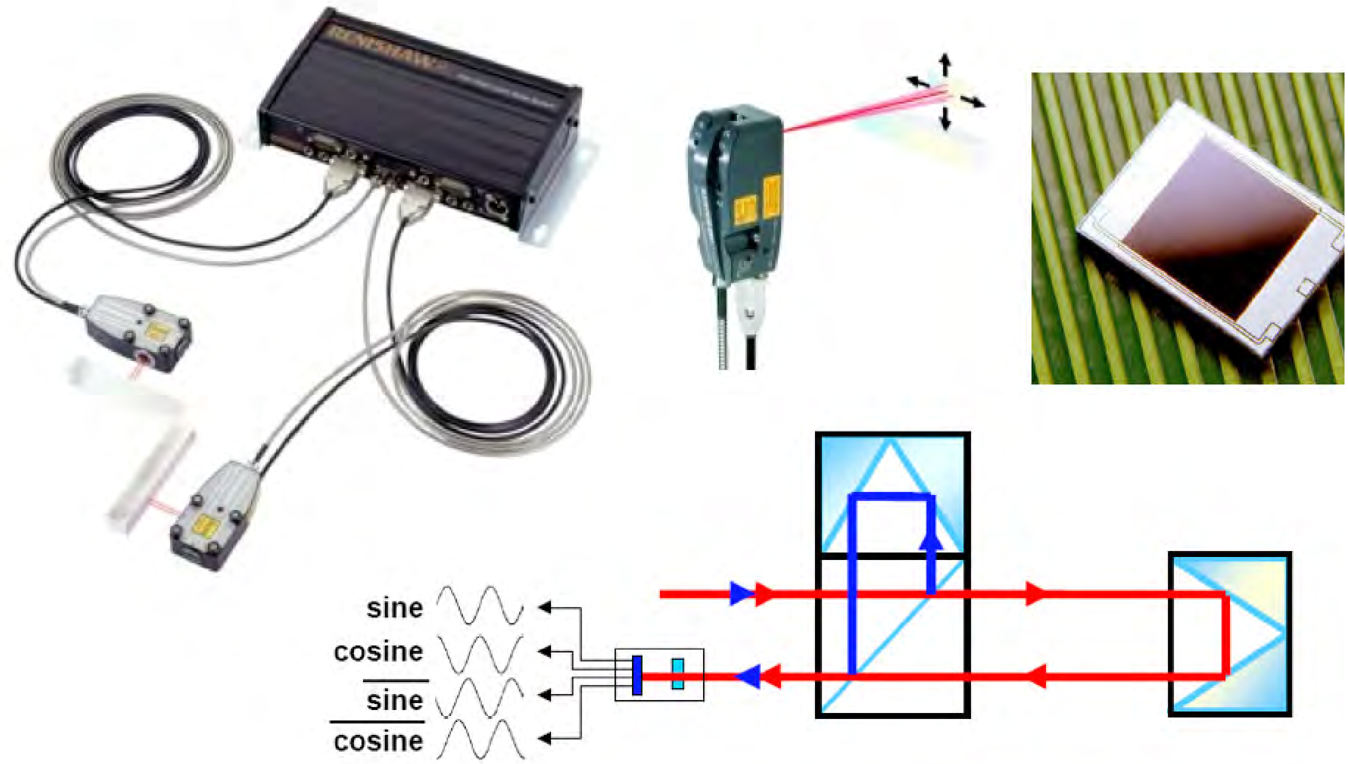

Figure 3. Laser interferometer system of the NIST Reference SEMs. Fiber-based beam delivery (upper left corner) Differential interferometer (upper center) Phase sensitive detector (upper right) and the detection scheme (lower center). (Courtesy of Renishaw Plc.)

the semiconductor industry. Reference Material 8120 line width samples will be a relevant sample on a 200 mm and 300 mm Si wafer with amorphous Si features with sizes from 1 mm to down to 50 nm. Standard Reference Material 2120 is going to be the calibrated, traceable version for line width measurements. This work is being carried out in cooperation with International SEMATECH.

Development of High Accuracy Laser Interferometer Sample Stage for SEMs: The development of a very fast, very accurate laser stage measurement system facilitates a new method to enhance the image and line scan resolution of SEMs. This method, allows for fast signal intensity and displacement measurements, and can report hundreds of thousands of measurement points in just a few seconds. It is possible then, to account for the stage position in almost real time with a resolution of 0.04 nm. The extent and direction of the stage motion reveal important characteristics of the stage vibration and drift, and helps minimize them. Figure 3 shows the key elements of the laser interferometer of the sample stage, the fiber laser beam delivery, the area detector, the interferometer and the detection scheme. The high accuracy and speed also allow for a convenient and effective technique for diminishing these problems by correlating instantaneous position and imaging intensity. The new measurement technique developed recently gives a possibility for significantly improving SEM-based dimensional measurement quality. Important new discoveries made it possible to correctly understand the motion of the stage at the nm level, and the characterization of various settings and fine tuning of the sample stage, which is critical for high-resolution work.
The laser interferometer sample stage and its control system designed and made by Fjeld Co. Figure 4 shows the essential components of a laser-interferometer-based SEM: the sample stage with a 300 mm wafer and the two fiber optics laser interferometers secured on the side and on the heavily reinforced door of the SEM.

The laser interferometer sample stage with suitable sample holder attachments can work with 200 mm and 300 mm wafers and 150 mm and 230 mm (six-inch and nine-inch) photomasks. These are designed to set the sample surface at four mm optimized working distance. A somewhat larger than 100 mm by 100 mm measurement coverage in the center allows for calibrations and measurements at the most ideal sample locations. The laser interferometer sample stage hardware and software have 38 pm resolution and

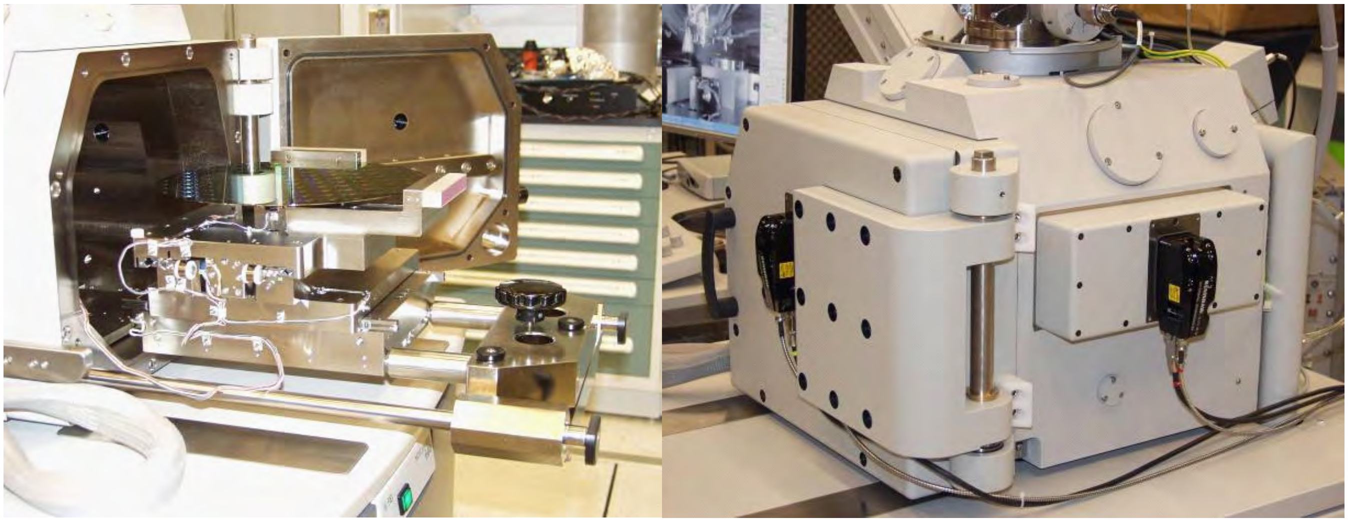

Figure 4. Essential components of a laser-interferometer-based SEM. The sample stage with a 300 mm wafer (left).The two fiber optics laser interferometers secured on the side and on the heavily reinforced door of the SEM (right).

very high read-out rates, which allow for drift and vibration compensation. This instrument is capable of measuring a very large and diverse set of samples, including samples without conductive coating. Figure 4 shows the laser interferometer sample stage, the high-precision exchange mechanism, and a 300 mm wafer mounted on the stage. The door is reinforced with a strong horizontal bar to minimize warping due to atmospheric changes. The Y direction laser interferometer is located behind the circular opening of the hinged door and the X direction laser is mounted on the right side of the sample chamber. The sample stage is shown in exchange position; it can slide in and get securely bolted down to the bottom of the sample chamber. The samples sit between the X and Y mirrors that -after sample exchange and a quick realignment- track the motion of the sample stage.

Development of Ultra-High Resolution He Ion Microscope (HIM): The HIM technology is not yet as optimized, developed or as mature as the SEM. As a new technique, HIM is just beginning to show promise and many potentially advantageous applications for integrated circuit and nanotechnology have yet to be exploited. Now that commercial instrumentation is available, further work is being done on the fundamental science of helium ion beam generation, helium ion beam-specimen interactions, and the signal generation and contrast mechanisms defining the image.

In addition to these areas of work, modeling needs to be developed to correctly interpret the signal generation mechanisms and to understand the imaging mechanisms. These are indispensable for accurate nanometer-level metrology. HIM and SEM have some overlapping territory, but they remain complementary techniques. Helium ion beam microscopy is forging into new scientific and technology territories, and this new and innovative technology will develop new science and contribute to the progress in integrated circuit and nanotechnology.

Beyond high-resolution imaging, He ion beam is also useful for exposition of resist materials. Figure 5 shows dense array of approximately 15 nm diameter hydrogen silsesquioxane (HSQ) resist posts generated by He ion lithography. Smaller structures are also feasible and the sensitivity of resists to ion irradiation can be significantly higher than to electrons. This are is also being pursued to investigate how far ion-beam lithography can be pushed.

Accomplishments

- Developed two Reference Metrology SEMs equipped with laser interferometry, ready for traceable calibrations of masks, wafers and other samples.
- Characterized all important SEM parameters to minimize measurement uncertainty.
- Developed quantitative nanoparticle size analysis and dimensional metrology methods.

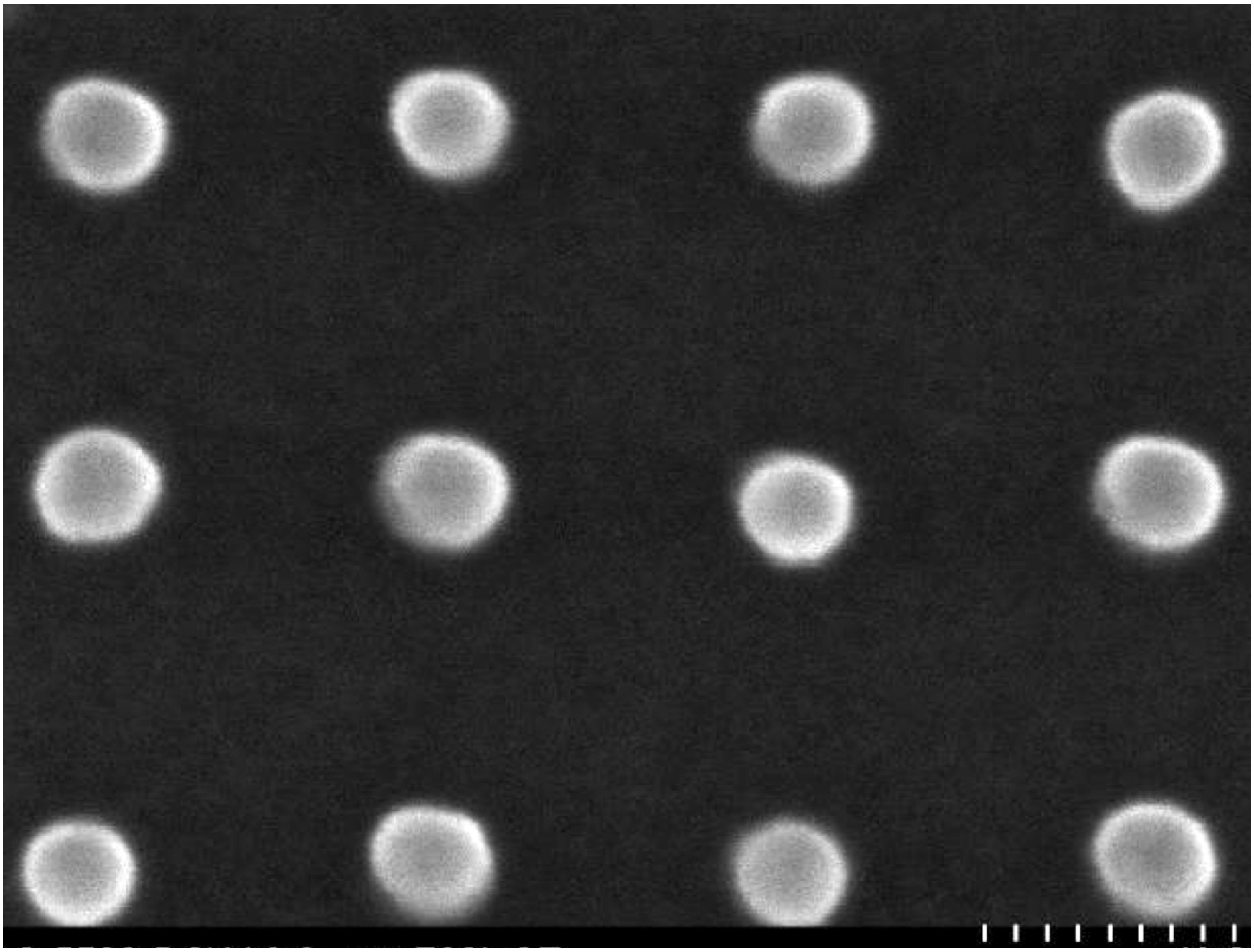

Figure 5. Dense array of 15 nm hydrogen silsesquioxane (HSQ) resist posts generated by He ion lithography. 180 nm field of view (right).

- Delivered four Reference Materials useful for a wide variety SEM, AFM and optical microscopy applications.

Impact/Benefit:

The project has significant impact and benefited the semiconductor manufacturing industry in the past many years through a close collaboration with International SEMATECH, and its Advance Metrology Advisory Group.

- A Photomask Metrology Project was carried out and established sound SEM, optical and scatterometry metrology for photomasks.
- Line Edge and Line Width Roughness Metrology Project was carried out and established sound SEM and scanning probe metrology methods, with significant technical papers published relevant conferences.
- A Project for the development of Reference Critical Dimension Metrology Scanning Electron Microscope was successfully carried out and finished in 2008.
- A Project on the development of sound metrology methods for contour measurements is underway in 2009.
- New significantly improved measurement methods for fast image collection were worked out and introduced to the IC industry.
- Cooperation with key SEM manufactures is also underway that help the widespread introduction the high-performance and highly accurate measuring methods developed at NIST in this project.
- An expanded measurement uncertainty statement that spells out all significant sources of measurement error was worked out. This could help the industry to carry out more accurate dimensional measurements.
- The applicability and various measurement and fabrication methods of HIM technology for the semiconductor industry are under investigation. The findings could help to introduce this new technology for

industrial use.

Publication List:

Book Chapters

- Vladar A. and Postek, M. T. 2009. The Scanning Electron Microscope. Handbook of Charged Particle Optics. (ed. Jon Orloff) CRC Press, Inc., New York. (437- 496).

Conference Proceedings

- Postek, M. T. and John A. Allgair. 2009. Instrumentation, Metrology, and Standards for Nanomanufacturing III. SPIE Volume 7042. SPIE Bellingham, Washington.

2009

- Postek, M. T. and Lyons, K. 2009. Instrumentation, metrology and standards, three keys to the nanotech kingdom. Micromanufacturing Proc. SPIE 7405 740503-1- 740503-18.
- Postek, M. T. and Brown, E. 2009. Sustainable, renewable nanomaterials may replace carbon nanotubes. SPIE Newsroom, 10.1117/2.1200903.1474.
- Obikawa, T., Postek, M. T.,Dornfeld, D., Liu, R., Komanduri, R., Guo, Y., Shi, J.,Cao, J., Zhou, J., Yang, X. and Xiaochun., X. 2009. LiMSEC 2009 State-of the art paper: Micro/Nanotechnology Applications for manufacturing systems and processes. Proceedings 2009 International Manufacturing Science and Engineering Conference (In press Published by ASME.).
- Lyons, K., and Postek, M. T. 2009. Historic commitment of the NIST Manufacturing Engineering Laboratory to nanomanufacturing and nanometrology. SPIE Proceedings. Proc. SPIE 7405 740503-1 – 740503-18.
- Vladar, A. E. and Postek, M. T. 2009. Reference Material (RM) 8820: A new scanning electron microscope scale standard. Microscopy and Microanalysis 2009 668-669..
- Stefaniak, A., Hackley, V., and Postek, M. T. 2009 Nanoscale reference materials for environmental, health and safety applications. 2009 4th International Helsinki Conference (in press).
- Decker, J., Hight-Walker, A., Bosnick, K., Clifford, C., Dai, L, Fagan, J., Hooker, S., Jakubek Z., Kingston, C., Makar, J., Postek, M.T., Simard, B., Sturgeon, R., Wise, S., Vladar, A., Yang, L., and Zeisler, R. 2009. Sample Preparation Protocols for Realization of Reproducible Characterization of Single-walled Carbon Nanotubes. NIST J. Res. (in review).
- Oral M.and A. E. Vladár "Optimization of SEM Imaging through 3D Modeling of Beam and Sample" SCANNING Meeting Monterey May 2009
- Cizmar,P., A. E. Vladár and M. T. Postek "Image Simulation for Testing of SEM Resolution Measurement" Methods SCANNING Meeting Monterey May 2009
- Silver R. and A. E. Vladár, EUV Metrology for EUV Lithography, McGraw Hill, 2009

2008

- Postek, M. T., K. Lyons, M. Uimette and G. Holdridge. 2008. Interagency Working Group Workshop Report on Instrumentation Metrology and Standards for Nanomanufacturing 2008. Interagency Working Group Workshop Report on Instrumentation Metrology and Standards for Nanomanufacturing 2008 131pp. available to download from www.manufacturing.gov.
- Postek, M. T., Vladar, A., Dagata, J.,Farkas, N., Ming, B.,and Wegner, T. 2008. Cellulose Nanocrystals the Next Big Nano-thing? Proc. SPIE 7042: 70420D-1 - 70420D-11.
- Lyons K. W. and Postek, M. T. 2008. Metrology at the nanoscale: What are the Grand Challenges? Proc. SPIE 7042 704202-1 – 704202-13
- Postek, M. T. et al. 2008. National Nanotechnology Initiative Supplement to the President's Budget 2008. Co-developed by the Nanoscale Science, Engineering and Technology Submmittee, Committee on Technology, National Science and Technology Council (M. Postek participant and contributor) download: http://www.nano.gov.
- Vladár A. and M. T. Postek "Development of Reference Critical Dimension Metrology Scanning Electron Microscope" SEMATECH Final Report. 2008
- Vladár, A., K. P. Purushotham and M. T. Postek "Contamination Specification for Dimensional Metrology SEMs" SPIE Microlithography 2008
- Postek, M., A. E. Vladár "The Potentials of Helium Ion Microscopy for Semiconductor Process Metrology" SPIE Microlithography 2008
- Vladár, A., J. S. Villarrubia, P. Cizmar, M. Oral and M. T. Postek "Accurate and Traceable Dimensional Metrology with a Reference CD-SEM" SPIE Microlithography 2008

2007

- Postek M. T. et al. 2007. The National Nanotechnology Initiative Research and Development Leading to a Revolution in Technology and Industry – Supplement to the President's FY 2007 Budget. Developed by the Nanoscale Science, Engineering and Technology Subcommittee, Committee on Technology, National Science and Technology Council (M. Postek participant and contributor) download: http://www.nano.gov.
- Postek M. T. et al. 2007. The National Nanotechnology Initiative Strategic Plan - December 2007. Developed by the Nanoscale Science, Engineering and Technology Subcommittee, Committee on Technology, National Science and Technology Council (M. Postek participant and contributor) download: http://www.nano.gov.
- Postek, M. T. and K. Lyons. 2007 Instrumentation, Metrology, and Standards Key Elements for the Future of Nanomanufacturing. SPIE Nanomanufacturing Conference 6648, pp 664802-1 - 664802-7.

2006

- Londoño, C., Lawall,J., Postek, M. T., Stone, J. and Stoup, J. 2006. The NIST Advanced Measurement Laboratory: At the Leading Edge of Measurement Science and Technology! Proceedings SPIE San Diego 6292. pp 629209-1 – 629209-5.
- Postek, M. T., Kopanski, J and Wollman, D. 2006. NIST Accomplishments in Nanotechnology. NIST Special Publication 1052, 186pp. and CD.
- Knight, S. Dixson, R., Jones, R., Lin, E., Orji, N., Silver, R., Villarrubia, J., Vladár A.and Wu W-L. 2006. Advanced metrology needs for nanoelectronics lithography, Besoins en métrologie avancée pour la lithographie nanoélectronique " Comptes Rendus Physique Volume 7, Issue 8, Pages 931-941.
- Postek, M. T. et al. 2006. National Nanotechnology Initiative Supplement to the President's Budget 2006. Developed by the Nanoscale Science, Engineering and Technology Subcommittee, Committee on Technology, National Science and Technology Council (M. Postek participant and contributor) download: http://www.nano.gov.
- Gorman, J., Kim Y-S., Vladar, A., Dagalakis, N, 2006. Design of an on-chip microscale nanoassembly system" International Journal of Nanomanufacturing Volume 1, Number 6.
- Postek, M. T. et al. 2006. National Nanotechnology Initiative. NNI Grand Challenge Workshop Report. Instrumentation and Metrology. (Michael Postek, Chairman) (August 2006 available at www.nano.gov).
- Bunday, B.. Banke, B., Archie, C. Solecky, E., Cao G., Hartig, G., Azordegan, A., Postek, M., Villarrubia, J., Vladár, A., Joy, D. and Singh, B. 2006. Unified Advanced Critical Dimension Scanning Electron Microscope (CD–SEM) Specification for sub-90 nm Technology (2005 Version) International SEMATECH Manufacturing Initiative Technology Transfer #04114595B-ENG. 1- 152pp.

2005

- Villarrubia, J. S., Vladar, A. E. and Postek, M. T. 2005. Scanning electron microscope dimensional metrology using a model-based library. Surface and interface Analysis 37:951-958.
- Postek, M. T., Villarrubia, J. and Vladar, A. 2005. Advanced Electron Microscopy Needs for Nanotechnology and Nanomanufacturing. J. Vac. Sci. Technol. B, Vol. 23, No. 6, Nov/Dec 2005 3015-3022.
- Villarrubia, J. S., Vladar, A. E. and Postek, M. T. 2005. A Simulation Study of Repeatability and Bias in the CD-SEM. J. Microlithography Microfabrication and Microsystems. 4(3) 033002-1 - 033002-10.
- Zhang, N. F, Vladar, A.E., Postek, M. T. and Larrabee, R. D. L. 2005. Spectral density-based statistical measures for image sharpness. Metrologia 42:351-359.
- Vladar, A. E., Postek, M. T. and Joy, D. C. 2005. Nano-tip Electron Gun for the Scanning Electron Microscope. MSA Proceedings Microscopy and Microanalysis (supp 2) 766 - 767.
- Tanaka, M., Villarrubia, J.S. Vladar, A. E. 2005. Influence of Focus Variation on Linewidth Measurements , Proceedings of SPIE, Feb. 27-March 4, 2005, San Jose, CA, 5752, 144-55,

Presentations:

Short Course

- Instructor in the Scanning microscopy in the Forensic Science Short Course held at the 2009 SPIE/ SCANNING meeting.

Conference Chair

- 2009 SPIE/SCANNING
- 2008 SCANNING
- SPIE Conference entitled "Instrumentation, Metrology and Standards for Nanomanufacturing III.
- SPIE Conference entitled "Instrumentation, Metrology

and Standards for Nanomanufacturing II.

- SPIE Conference entitled "Instrumentation, Metrology and Standards for Nanomanufacturing I.

Other

- SME Video Communications Department produced a new 30 minute 'Manufacturing Insights' video program on Nanometrology Michael Postek was invited to provide expert commentary on the field of Nanometrology for this program.

Invited and Contributed

- "Simulation Study for CD-SEM," J. S. Villarrubia, SEMATECH Advanced Metrology Advisory Group meeting, Albany, NY, September 10, 2009.
- "Measurement Science Addressing the EH&S Aspects of Engineered Nanomaterials "Tiny Threats" by the International Life Sciences Institute (ILSI). 2009. Tuscon, AZ".
- "Measurements, Instrumentation and Standards for Nanotechnology." Society of Manufacturing Engineers, 2008, Second Manufacturing Education Leadership Forum Long Island, NY.
- "Nanotechnology: Important link between trees and airplanes." 2008 Forest Products Industry National Meeting St. Louis MO.
- "You Can't Make It if You Can't Measure It." SME 2007 Meeting Boston, MA
- "Simulation Study for Electron Beam Inspection Tools," J. S. Villarrubia, SEMATECH Defect Metrology Advisory Group meeting, Albany, NY, September 10, 2009.
- Invited presentation at the Nanomanufacturing Summit 2009 "Measurements Instrumentation and Standards for Nanotechnology."
- NIOSH, Morgantown: "Challenges for nanoparticle characterization."
- Advanced Curriculum Working Group: Nanotechnologies. Society of Manufacturing Engineers Second Manufacturing Education Leadership Forum, Long Island, NY.
- "Cellulose Nanocrystals the Next Big Nano-thing?" SPIE Instrumentation, Metrology and Standards for Nanomanufacturing. August 2008 San Diego, CA.
- "Nanotechnology Directions within the Dimensional and Mechanical Metrology Programs of the NIST Manufacturing Engineering Laboratory" for the Tri-National Workshop held at NIST
- "Key Elements for the Future of Nanomanufacturing: Instrumentation, Metrology, and Standards" at a workshop hosted by the Center for Hierarchical Manufacturing, an NSF Nanoscale Science & Engineering Center
- "Nanometrology: A Key Element for Successful Nanomanufacturing" 2007 AVS Meeting October 2007, Seattle, WA
- "New NIST Reference Material RM 8475: Carbon, Metal Catalyst and Carbon Nanotubes. NSTI Conference, Santa Clara, CA.
- "Documentary and Artifact Standards Activities for Nanotechnology" – ASME B-89 Meeting, Charlottte, NC.2007 "Helium Ion Microscopy: a New Technique for Semiconductor Metrology and Nanotechnology" was presented at the Frontiers of Characterization and Metrology for Nanoelectronics Conference Gaithersburg, MD, February 2007.
- "NIST: The Nanometrology Resource for Nanotechnology and Nanomanufacturing" at the SEMI NanoForum 2006 meeting in San Jose, California, November, 2006
- "Accurate Measurements for Nanotechnology and Nanomanufacturing: Are they Possible?" Hitachi High Technologies America (HHTA) at their National Nanotechnology Seminar Series held at the NASA-AMES Facility at Moffett Field, CA., November, 2006
- "Introduction and Review of the Research Needs Identified in the 2004 Instrumentation and Metrology Workshop" Instrumentation, Metrology, and Standards for Nanomanufacturing a Workshop of the National Science and Technology Council (NSTC) Interagency Working Group (IWG) on Manufacturing Research and Development (R&D) October 2006.
- "Infrastructure needs for nanotechnology" NNI 2009 Regional, State, & Local Initiatives in Nanotechnology Workshop - Invited Panel Member, Oklahoma City.
- "Nanometrology and Nanomanufacturing at the National Institute of Standards and Technology" The 2006 Micro Nano Breakthrough Conference "Building a Micro/Nano Tech Economy" hosted by ONAMI, Vancouver WA, July 2006.
- "Nanometrology and Nanomanufacturing at the National Institute of Standards and Technology" Boeing Company Seattle, WA, July 2006.
- "Nanometrology and Nanomanufacturing at the National

Institute of Standards and Technology" Oregon State University, Eugene, Oregon, July 2006.

- "NIST Nanotechnology" in the Nanotechnology and Advanced Materials session led by Dr. Arden Bement of NSF of the US Department of Commerce and Japan's Ministry of Economy, Trade and Industry (METI) held a meeting at the National Science Foundation. Arlington, VA, May 2006.
- "Nanotechnology at the National Institute of Standards and Technology" in the Overview of US Government R&D Programs on Nanotechnology session at the INC2 2006 meeting (Second International Nanotechnology Conference on Communication and Cooperation) in Crystal City, VA, May 2006.
- "SEM-based Dimensional Metrology at the Nanometer Scale" A. E. Vladar, M. T. Postek, J. S. Villarrubia SEM Based Dimensional Metrology Braunschweig, Germany 2006
- "Nanotechnology Research at the National Institute of Standards and Technology" to the NSET Subcommittee of the NNI, May 2006.
- "NIST Nanotechnology and Nanomanufacturing" at the National Research Council of Canada Institute for National Measurement Standards (NRC-INMS) Ottawa Canada, March 2006.
- "NIST Nanotechnology and Nanomanufacturing" at Albany Nanotech, Albany NY, March 2006.
- "NIST: The Nanometrology Resource for Nanotechnology" Drexel University Philadelphia, PA, December 2005.
- "Advanced Scanning Electron Microscopy Needs for Nanotechnology and Nanomanufacturing" Nano 2005 Boston, MA November 2005.
- "Unique Nanometrology Research Capabilities at NIST" Maryland Department of Business and Economic Development (DBED)/Univ. of Maryland and IMEC Event, College Park, MD 2005.
- "NIST: The Nanometrology Resource for Nanotechnology" Microsystems and Nano Economic Summit, Sponsored by the OhioMEMS Association in Cleveland Ohio. September 2005.
- "Nanotechnology R&D at the National Institute of Standards and Technology" Future Manufacturing Forum at the Mont Royal Centre in Montreal, Canada, September 2005.
- "Nanotechnology Research and Development at NIST at the Workshop on Nanotechnology Applications to Chemical and Biological Defense and Homeland Security." Workshop on Nanotechnology Applications to Chemical and Biological Defense and Homeland Security, Reston VA, September 2005.
- "NIST: Nanometrology Resource for Biomedical Applications of Nanotechnology." 2005 US Measurement System Workshop on Nanobiotechnology (held during the American Chemical Society Meeting at the Washington D.C. Convention Center August 2005.
- "Metrology Research in the NIST Advanced Measurement Laboratory." NCSL Workshop Gaithersburg, MS August 2005.
- "Variable Pressure/Environmental SEM a Powerful Tool for Nanotechnology and Nanomanufacturing" Microscopy Society of America Hawaii 2005.
- "Metrology for a New Science: Advanced Metrology Needs for Nanotechnology and Nanomanufacturing" Micro Nano Breakthrough Conference, Portland OR July 2005.
- "Variable Pressure/Environmental Scanning Electron Microscopy: Application to Photomask Dimensional Metrology" Micro Nano Breakthrough Conference, Portland OR July 2005.
- "Advanced Metrology Needs for Nanotechnology and Nanomanufacturing" at the Electron, Ion and Photon Beam Technology and Nanofabrication Meeting in Orlando Florida. June 2005
- "Metrology: Fundamental for Realizing Products at the Nanoscale" The Society of Manufacturing Engineers Minneapolis, MN April 2005.
- NIST Nanotechnology overview at the DOE/NIBIB Workshop on Biomedical Applications of Nanotechnology in Bethesda, MD March 2005.
- "NNI Grand Challenge Instrumentation and Metrology Workshop" Nanotechnology in Microlithography Technical Working Group Meeting at the 2005 SPIE Microlithography meeting San Jose, Calif. March 2005.
- "SEM Modeling for Dimensional Metrology," J. S. Villarrubia, SEMATECH Advanced Metrology Advisory Group meeting, Monterey, CA, February 19, 2009.
- "SEM Modeling for Defect Metrology," J. S. Villarrubia, SEMATECH Defect Metrology Advisory Group meeting, Monterey, CA, February 19, 2009.
- "SEM Modeling for Defect Metrology" SEMATECH Defect Metrology Advisory Group meeting, Austin, TX,

Sept. 24, 2008.

- "SEM Modeling for Metrology" J. S. Villarrubia, SEMATECH Advanced Metrology Advisory Group meeting, Austin, TX, Sept. 24, 2008.
- "Applications of Mathematical Morphology to AFM," SCANNING, Gaithersburg, MD, April 17, 2008.
- "E-Beam/Sample Interaction Modeling," SEMATECH Advanced Metrology Advisory Group, Monterey, CA, Feb. 21, 2008.
- "E-Beam Modeling for 3-D Samples," presented to SEMATECH Advanced Metrology Advisory Group, Austin, TX, Sept. 25, 2007.
- "Informatics Issues in Library-Based Dimensional Metrology," at Nanoinformatics Strategies, a NSF National Nanomanufacturing Network workshop, Arlington, VA, June 13, 2007.
- "Monte Carlo modeling of secondary electron imaging in three dimensions," SPIE Microlithography meeting, San Jose, CA, Feb. 28, 2007.
- "SEM and AFM modeling in 3-Dimensions," presented to SEMATECH Advanced Metrology Advisory Group, Santa Cruz, CA, Feb. 22, 2007.
- "Modeling for Dimensional Metrology using SecondaryElectron Scanning Electron Microscopy," Colloquium at SUNY Albany's College of Nanoscale Science and Engineering, Dec. 8, 2006.
- "Issues and Approaches in CD Metrology with AFM and SEM," Micro and Nano Technology Measurement Club, London, England, September, 20, 2006.
- "Model-Development for End of the Roadmap SEM Metrology," SEMATECH Metrology Council, Austin, TX, September 28, 2004.
- "Unbiased Estimation of Linewidth Roughness," SEMATECH Metrology Council, Austin, TX, February 23, 2005.
- "Metrology Issues in LER and LWR," Hitachi Technical Forum, San Jose, CA, March 2, 2005.
- "SEM Dimensional Metrology Using a Model-Based Library," International Workshop on Modeling Electron Transport for Applications in Electron and X-Ray Analysis," Gaithersburg, MD, November 9, 2004.
- "Issues in Line Edge and Linewidth Roughness Metrology," International Conference on Characterization and Metrology for ULSI Technology, Richardson, TX, March 16, 2005.

Contributed

- "SEM Modeling for Dimensional Metrology," J. S. Villarrubia, NIST Precision Engineering Division Seminar, Gaithersburg, MD, March 25, 2009.
- "Sensitivity of SEM width measurements to model assumptions," J. S. Villarrubia, SPIE Advanced Lithography, San Jose, CA, February 24, 2009.
- "Collaboration Opportunities in SEM modeling," a presentation to selected faculty of the College of nanoscale Science and Engineering at the Albany Nanotech center, Albany, NY, June 10, 2008.
- "Physics for Secondary Electron Generation," SCANNING, Monterey, CA, April, 12, 2007.
- "High Energy Electron/Solid State Interaction Modeling with MONSEL," Victor Katsap and John Villarrubia, International Vacuum Electronics Conference, Monterey, CA, April 25-27, 2005.
- "Unbiased Estimation of Linewidth Roughness," presented at SPIE conference on Metrology, Inspection, and Process Control, San Jose, CA, March 2, 2005.

Awards:

- Silver medal for development of model-based library method for SEM, awarded by Dept. of Commerce.
- Diana Nyyssonen Metrology Best Paper of 2005 for "Unbiased Estimation of Line-Edge and Line-Width Roughness," awarded by SPIE Advanced Lithography Conference on Metrology, Inspection, and Process Control.

Customers:

- International SEMATECH and its member companies
- CD-SEM and other SEM instrument manufacturers
- Users of various standard methods and artifacts across the world

Collaborators:

- International SEMATECH, Advanced Metrology Advisory Group
- International Technology Roadmap for Semiconductors; Microscopy and Metrology Sections
- Zeiss/ALIS Corp.
- FEI Co.
- ISO
- E. Fjeld Co.

For Further Information Contact:

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Wafer Level SEM Metrology for Critical Dimension Measurements: Modeling

Industry Need:

"Stack materials, surface condition, line shape and even layout in the line vicinity may affect CD-SEM waveform and, therefore, extracted line CD. These effects, unless they are accurately modeled and corrected, increase measurement variation and total uncertainty of CD SEM measurements."—International Technology Roadmap for Semiconductors, Metrology Section, p. 10 (2007).

"A better understanding of the relationship between the physical object and the waveform analyzed by the instrument is expected to improve CD measurement."—International Technology Roadmap for Semiconductors, Metrology Section, p. 8 (2007).

"Due to the changing aspect ratios of IC features, besides the traditional lateral feature size (for example, linewidth measurement) full three-dimensional shape measurements are gaining importance and should be available inline." —International Technology Roadmap for Semiconductors, Metrology Section, p. 8 (2007).

A feature's width is one of its fundamental dimensional characteristics. Width measurement is important in a number of industries including the semiconductor electronics industry, with worldwide sales of $248.6 billion in 2008 [Semiconductor Industry Association].

To support present and future semiconductor technologies, industry needs to measure gate electrode widths with total uncertainties, as identified in the International Technology Roadmap for Semiconductors (ITRS, 2008 update), of approximately 0.42 nm. In addition to measuring linewidths, the semiconductor industry has needs for measuring linewidth variation, that is, linewidth roughness (LWR). LWR in transistor gates has been linked to increased off-state leakage current and to threshold voltage variation. The 2008 ITRS update specifies that LWR, measured as three standard deviations of the critical dimension (CD), must be less than 2.12 nm in 2009 and be measured with no more than 0.42 nm uncertainty. Contour metrology is an emerging industry practice. A contour measurement consists of assigning a 2-dimensional object boundary in a top-view image (Fig. 1). Width, linewidth, and boundary contours must generally be determined from an object's image. However, the image is not an exact replica of the line. The scanning electron microscope (SEM), scanning probe microscope (SPM), and optical microscope all have image artifacts that are important at the relevant size scales. These artifacts are related to the probe-sample interaction physics and often result in systematic edge assignment errors. These errors

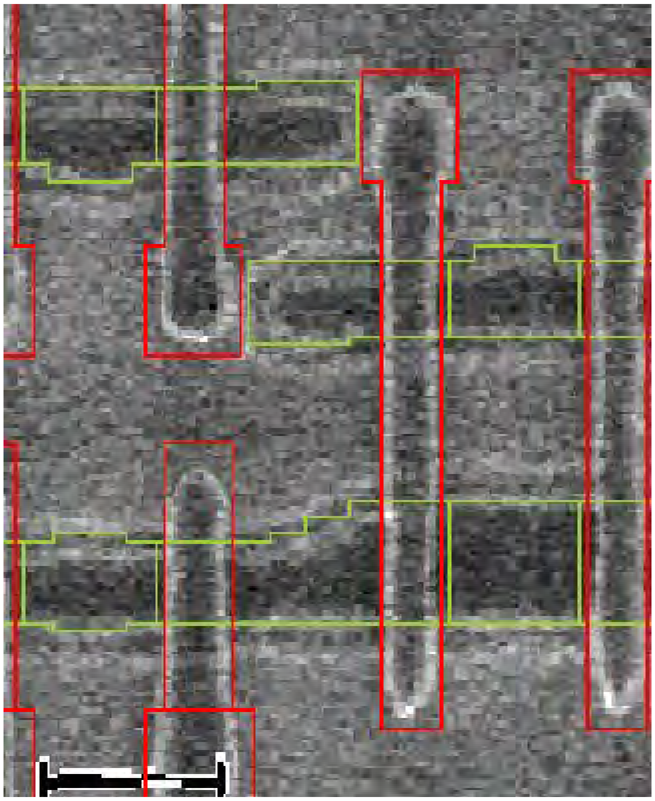

Figure 1. Example of contour metrology. Edge positions are assigned not only at a single position, but along full 2-D contours. (Image courtesy of Ben Bunday, ISMI)

are often not constant in a measurement comparison, but may vary due to details of the sample shape, instrument condition, the proximity of neighboring features, charging, contamination, etc. Determinations of width, roughness, and contour position therefore require modeling of the probe/sample interaction in order to correct image artifacts and identify edge locations.

Project Objective:

The goal of this project is to (1) develop and validate instrument function models for scanning electron microscopes and atomic force microscopes, (2) use those models to analyze existing measurement practices, alert industry metrologists to significant unrecognized errors, and suggest measurement practices less subject to error, (3) use the models to correct images for measurement artifacts, thereby obtaining improved accuracy.

Technical Approach

The main work of the project lies in the development of an accurate simulation tool for SEM. This tool is a computer program that calculates $M(x,y,S,p_i)$ where M is the calculated model intensity at position x, y produced by the SEM, S represents a description of the sample shape, and p_i represents all other input parameters, including those that describe the sample composition, beam shape, beam size, landing energy, etc. If the model is accurate and the inputs S and, p_i are set to the true values, then apart from random errors (noise) in the measured image, $I(x,y)$, we would have $M(x,y,S,p_i) = I(x,y)$. In practice, $I(x,y)$ and many instrument parameters are known, but the true sample geometry, S, and perhaps some other instrument parameters are unknown. Therefore we wish to invert the model function to obtain S and other unknowns as a function of the measured image and known parameters. For the SEM, M is usually in part a computationally intensive Monte Carlo calculation for which there is no simple inverse function. The early work of the project involved development of a model-based library (MBL, with concept shown in Fig. 2) inversion method.

> **"A better understanding of the relationship between the physical object and the waveform analyzed by the instrument is expected to improve CD measurement."**

In this method, the sample geometry is parameterized and M is tabulated for discrete parameter values within a range of interest. Then M can be approximated by interpolation and the parameters that best fit a given measurement can be determined by nonlinear least squares fitting methods. The model must account for all significant sources of topographic and material contrast in the measured image.

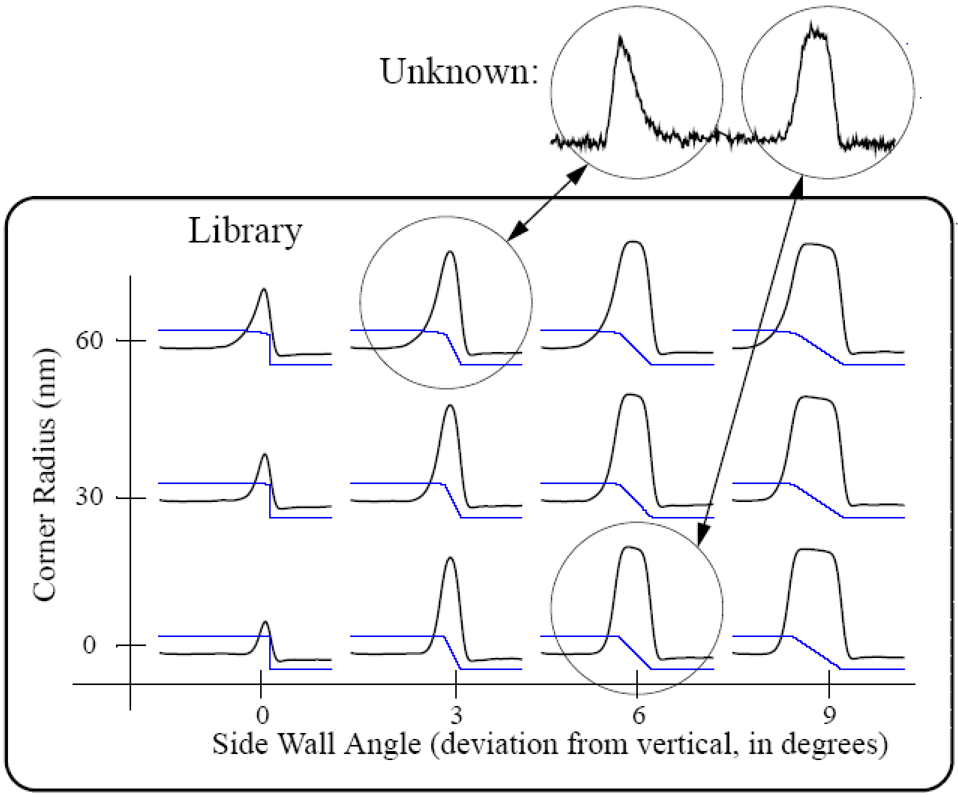

Figure 2. Schematic of model-based library metrology. Measured intensity profiles are matched to modeled profiles in a "library" or database. In practice the library is interpolated to improve the match. Parameters of the best matching modeled structure are attributed to the measured one.

For reasons of signal and throughput, the semiconductor industry mainly uses secondary electron images. The main processes that contribute to such images are beam formation (which determines beam size, beam shape, and depth of field), elastic scattering (which largely determines the shape of the scattering volume within the sample), inelastic scattering (determines the range of electrons and hence the size of the scattering volume, the intensity and initial trajectory of secondary electrons, and the secondary electron cascade), refraction or reflection at material boundaries, and transport, collection and detection of escaped secondary electrons. To perform an MBL measurement, the model must be applicable to the relevant measurement parameters (e.g., it must be able to simulate the actual sample shape and composition and others close to it). Accuracy of the model is also important. Improvements in model accuracy reduce measurement errors.

At the beginning of the reporting period, we were using the MONSEL (MONte Carlo simulator for Secondary ELectrons) simulator for MBL simulations. During this period we developed a replacement, JMONSEL (Java MONSEL), that retains all of MONSEL's capabilities while improving upon it in two important areas.

MONSEL was developed mainly for linewidth metrology. The sample shapes that could be simulated were restricted to particular parameterized classes. For example, one version could simulate 3 lines of uniform cross-section on a 3-layer substrate, with user settable linewidth, line spacing, line height, layer thicknesses, sidewall angles, etc. JMONSEL replaces this sample description with a constructive solid geometry (CSG) description. In CSG, 3-dimensional samples are described in terms of primitive building blocks (sphere, cylinder, convex polyhedra) of variable size and orientation. The building blocks can be combined by set operations (union, intersection,…) to form more complicated shapes. This description is considerably more flexible, permitting 3-D samples of virtually any shape to be approximated with any desired fidelity (Fig.3).

We have also been improving the model physics. The original models were binary scattering models. However, in condensed matter at energy exchange below about 100 eV, collective screening effects are important. We are in the process of implementing a model of inelastic electron

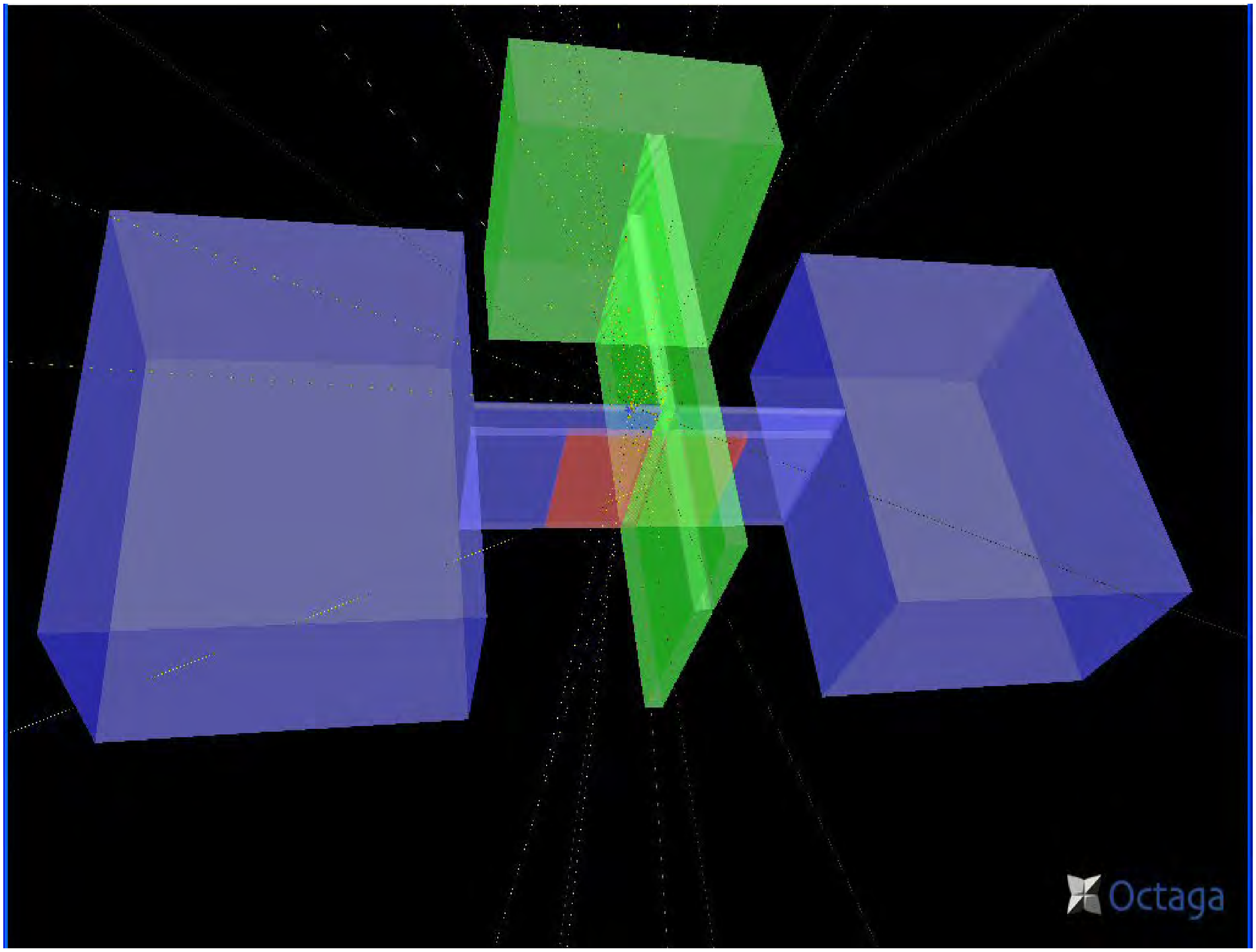

Figure 3. Model FinFET and simulated electron trajectories rendered in JMONSEL.

scattering based upon many-body dielectric function theory. This model requires scattering tables to be pre-computed for each material in the simulation. Material properties, in the form of a measured “energy loss function” equal to: Im [-1/ε (ω)] are required for each material. The necessary data for many materials are available at NIST because they were used to compute inelastic mean free paths for a NIST standard reference database (NIST SRD71). Our original implementation of this improved model was for Cu. An example of the agreement between measured and modeled Cu secondary electron energy spectra is shown in Fig. 4. The library of materials for which scattering tables are available has been expanded now to include 14 others. The project has financial support from SEMATECH to further expand the available materials to include many of relevance to the semiconductor industry. A model previously used by David Joy (U. Tenn. and ORNL) has been adapted for use with general CSG-represented samples. This model or MONSEL’s original binary model may serve as a back-up to simulate materials for which energy loss function input data are not available. Improvements have also been made to the model for scattering at material interfaces.

The project also has some activity in addressing geometrical probe-sample interactions for scanning probe microscopes (e.g., atomic force microscopy). Prior to the current reporting period, we demonstrated that this interaction is described by the mathematical morphology dilation operation and that the best surface reconstruction if the tip shape is known is an erosion. If a suitable sample of known shape is available for use as a tip characterizer, an outer bound can be placed on the tip shape by using erosion. We also proposed and validated a method, blind reconstruction, that can be used with sharp tip characterizers of un-measured shape. The theory and software to implement dilation, erosion, and blind reconstruction were published and have been extensively cited in the SPM literature. However, although this published theory is applicable to images, samples, and tips of any shape, the software implementation was restricted to those that could

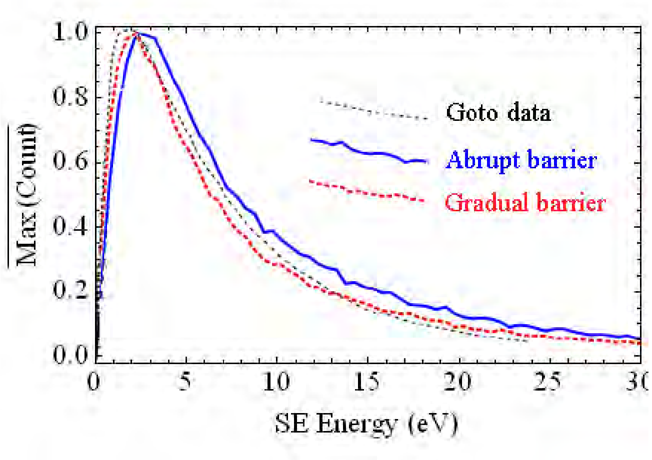

Figure 4. Secondary electron spectrum from 1 keV electrons incident on Cu as measured [Goto et al., Surf. Sci. 47, p. 477 (1975)] and as calculated in JMONSEL using the dielectric function theory model with two different barrier assumptions.

be represented by height maps (e.g., gray-scale images or single-valued functions). This was not a serious limitation at the time because most instruments used one-dimensional feedback that prevented them from making effective use of samples or tips that did not meet this restriction.

Since then, however, the use of "CD-AFM" instruments with boot-shaped tips and 2-dimensional feedback to scan samples with near-vertical or even undercut side-walls has grown, particularly in semiconductor and calibration applications where such instruments may serve as reference metrology tools. Accordingly, within the last few years we have collaborated with researchers at the Illinois Institute of Technology to extend the software capabilities to encompass this application. For this purpose, images, samples, and tips are represented by "dexels" rather than pixels. Dexels may have multiple heights in place of a pixel's single height, thereby allowing reentrant features to be represented. Dexel analogs of the aforementioned software algorithms have been developed, so it is now possible to perform image simulation, sample reconstruction, and tip reconstruction (blind or otherwise) for such objects (Fig. 5). This work is continuing with the development of a multi-dexel representation, the purpose of which is to describe complex samples with high fidelity without excessive use of computer memory.

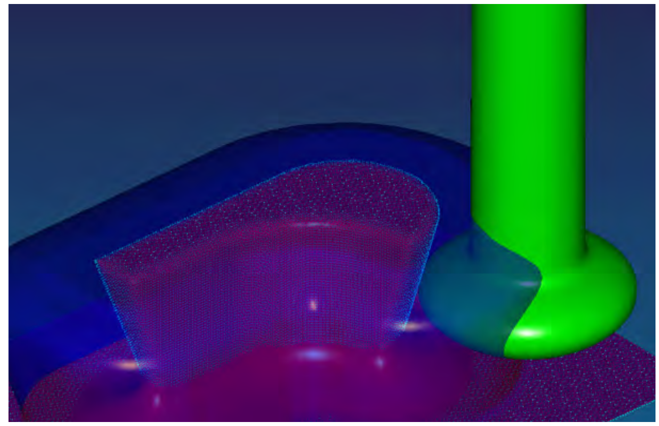

Figure 5. Sample (red) reconstruction from the image (blue) using a known tip (green) where all these objects are reentrant and described using dexels.

Impact/Benefit:

- SEM models are still at a research stage of development and validation, so are not yet routinely used by industry for metrology. However, as research tools our SEM modeling codes have already been used by at least 5 instrument suppliers (including 3 CD-SEM manufacturers), 5 semiconductor electronics manufacturers, and 7 universities or research institutes. Users do not always reveal their purposes to us, but uses we know about include assessing dimensional metrology errors, understanding unwanted backscatter exposure of electron beam resists in e-beam writers, evaluating the limits of SEM metrology, finding fast approximate models for SEM, and academic interests relating to secondary electron production.

- Prior to 2005, CD-SEM tools estimated linewidth roughness by calculating a multiple of the standard deviation of the linewidth variation. In a 2005 publication, we demonstrated to CD-SEM manufacturers and their customers that this is a biased estimate, and we demonstrated an alternative estimator that is unbiased. Today, all major CD-SEM suppliers claim unbiased roughness estimators for their instruments.

- SPM sample and tip reconstruction methods developed and published by this project have been cited over 170 times in the literature, have been incorporated into commercial software, and have been used in diverse applications including critical dimensions metrology, nanoscale mechanical characterization by nanoindentation, characterization of biological materials, surface roughness metrology, studies of crystallization and melting in polymers, tribology, and many others.

Accomplishments:

- Developed and published an unbiased linewidth roughness metric. Subsequently developed and published (with Applied Materials) a variant of the method that can be used with lines that shrink during measurement, as photoresist does.
- Wrote an SEM simulator that accepts samples of arbitrary 3-D shapes.
- Implemented (archival publication, with Illinois Institute of Technology) SPM image simulation, sample reconstruction, and blind tip estimation for reentrant samples and tips by using a new dexel representation for 3-D objects.
- Implemented a quantum mechanical model of electron scattering at a variable width and height potential energy step between materials (or the sample and vacuum).
- Developed a Monte Carlo implementation of a dielectric function theory (DFT)-based model of secondary electron (SE) generation. This model now includes the effects of screening, and computed inelastic mean free paths are in good agreement with NIST's standard reference database for inelastic mean free paths.
- Collected measured energy loss function (ELF) data for 40 elements and 17 compounds. These are the input data for computing scattering tables that are needed before a material can be modeled with the DFT model.
- Calculated DFT-model scattering tables for 14 materials.
- Implemented a phenomenological SE generation model with two fitting parameters. Materials for which the more detailed and accurate DFT-based model cannot be used because of missing material properties information can be fit with this back-up model.
- Implemented a model for electron-phonon scattering. This is especially important to model large band gap insulators like those (e.g., PMMA and SiO2) used heavily in semiconductor electronics applications.
- Studied (with archival publication) the sensitivity of SEM dimensional determinations to the choice of model. This study helped to identify which model uncertainties are especially important for dimensional measurements.

Publication List:

2009

- Villarrubia, J. S. and Ding, Z. J. 2009. Sensitivity of SEM width measurements to model assumptions. J. Micro/Nanolith. MEMS MOEMS 8, 033003.
- Villarrubia, J. S. and Ding, Z. J. 2009. Sensitivity of SEM width measurements to model assumptions. Proc. SPIE 7272, 72720R.

2008

- Tian, F., Qian, X. and Villarrubia, J. S. 2008, Blind estimation of general tip shape in AFM imaging. Ultramicroscopy 109, pp. 44-53.
- Vladar, A. E., Villarrubia, J. S., Cizmar, P., Oral, M. and Postek, M. T. 2008. Accurate and Traceable Dimensional Metrology with a Reference CD-SEM. Proc. SPIE 6922, 69220H.

2007

- Villarrubia, J. S., Ritchie, N. W. M. and Lowney, J. R. 2007. Monte Carlo modeling of secondary electron imaging in three dimensions. Proc. SPIE 6518, 65180K.
- Qian, X., Villarrubia, J., Tian, F. and Dixson, R. 2007. Image Simulation and Surface Reconstruction of Undercut Features in Atomic Force Microscopy. Proc. SPIE 6518, 651811.
- Wang, C., Jones, R. L., Lin, E. K. Wu, W., Villarrubia, J. S., Choi, K., Clarke, J. S., Rice, B. J., Leeson, M., Roberts, J., Bristol, R. and Bunday, B. D. 2007. Line-edge roughness and cross-sectional characterization of sub-50-nm structures using CD-SAXS: Round-robin benchmark results. Proc. SPIE 6518, 65181O.

2006

- Qian, X. and Villarrubia, J. S. 2007. General Three-Dimensional Image Simulation and Surface Reconstruction in Scanning Probe Microscopy using a Dexel Representation. Ultramicroscopy 108, pp. 29-42. doi:10.1016/j.ultramic.2007.02.031.
- Knight, S., Dixson, R., Jones, R. L., Lin, E. K. Orji, N. G., Silver, R., Villarrubia, J. S., Vlad‡r, A. E., Wu, W.-L. 2006. Advanced metrology needs for nanoelectronics lithography. C. R. Physique 7.
- Katz, R., Chase, C. D., Kris, R., Peltinov, R., Villarrubia, J. and Bunday, B. 2006. Bias reduction in roughness measurement through SEM noise removal. Proc. SPIE 6152, 61524L.

2005

- Postek, M. T., Villarrubia, J. S. and Vlad‡r, A. E. 2005. Advanced Metrology Needs for Nanotechnology and Nanomanufacturing. J. Vac. Sci. Technol. B 23, 3015-3022.

- Villarrubia, J. S. 2005. Issues in Line Edge and Linewidth Roughness Metrology. in Characterization and Metrology for ULSI Technology: 2005, AIP Conference Proceedings Vol. 788, edited by D. G. Seiler, A. C. Diebold, R. McDonald, C. R. Ayre, R. P. Khosla, S. Zollner, and E. M. Secula (AIP Press, New York) p. 386.
- Tanaka, M, Villarrubia, J. S. and Vladar, A. V. 2005. Influence of Focus Variation on Linewidth Measurements. Proc. SPIE 5752 pp. 144-155.
- Villarrubia, J. S. and Bunday, B. D. 2005. Unbiased Estimation of Linewidth Roughness. Proc. SPIE 5752 pp. 480-488.
- Villarrubia, J. S., A. E. Vlad‡r, and Postek, M. T. 2005. A Simulation Study of Repeatability and Bias in the CD-SEM. J. Microlith., Microfab., Microsyst. 4, 033002-1 through 033002-10.
- Villarrubia, J. S., A. E. Vlad‡r, and Postek, M. T. 2005. Scanning electron microscope dimensional metrology using a model-based library. Surf. Interface Anal. 37, 951-958.

Presentations:

Short Course

- “Interconnect-Relevant Characterization and Metrology Techniques,” a 1-day short course for attendees of the Advanced Metallization Conference in San Diego, CA, October 16, 2006

Panel Discussion

- “Metrology and Processing for Advanced Metallization panel,” at the Advanced Metallization Conference in San Diego, CA, October 16, 2006.

Invited

- “Simulation Study for CD-SEM,” J. S. Villarrubia, SEMATECH Advanced Metrology Advisory Group meeting, Albany, NY, September 10, 2009.
- “Simulation Study for Electron Beam Inspection Tools,” J. S. Villarrubia, SEMATECH Defect Metrology Advisory Group meeting, Albany, NY, September 10, 2009.
- “SEM Modeling for Dimensional Metrology,” J. S. Villarrubia, SEMATECH Advanced Metrology Advisory Group meeting, Monterey, CA, February 19, 2009.
- “SEM Modeling for Defect Metrology,” J. S. Villarrubia, SEMATECH Defect Metrology Advisory Group meeting, Monterey, CA, February 19, 2009.
- “SEM Modeling for Defect Metrology” SEMATECH Defect Metrology Advisory Group meeting, Austin, TX, Sept. 24, 2008.
- “SEM Modeling for Metrology” J. S. Villarrubia, SEMATECH Advanced Metrology Advisory Group meeting, Austin, TX, Sept. 24, 2008.
- “Applications of Mathematical Morphology to AFM,” SCANNING, Gaithersburg, MD, April 17, 2008.
- “E-Beam/Sample Interaction Modeling,” SEMATECH Advanced Metrology Advisory Group, Monterey, CA, Feb. 21, 2008.
- “E-Beam Modeling for 3-D Samples,” presented to SEMATECH Advanced Metrology Advisory Group, Austin, TX, Sept. 25, 2007.
- “Informatics Issues in Library-Based Dimensional Metrology,” at Nanoinformatics Strategies, a NSF National Nanomanufacturing Network workshop, Arlington, VA, June 13, 2007.
- “Monte Carlo modeling of secondary electron imaging in three dimensions,” SPIE Microlithography meeting, San Jose, CA, Feb. 28, 2007.
- “SEM and AFM modeling in 3-Dimensions,” presented to SEMATECH Advanced Metrology Advisory Group, Santa Cruz, CA, Feb. 22, 2007.
- “Modeling for Dimensional Metrology using SecondaryElectron Scanning Electron Microscopy,” Colloquium at SUNY Albany’s College of Nanoscale Science and Engineering, Dec. 8, 2006.
- “Issues and Approaches in CD Metrology with AFM and SEM,” Micro and Nano Technology Measurement Club, London, England, September, 20, 2006.
- “Model-Development for End of the Roadmap SEM Metrology,” SEMATECH Metrology Council, Austin, TX, September 28, 2004.
- “Unbiased Estimation of Linewidth Roughness,” SEMATECH Metrology Council, Austin, TX, February 23, 2005.
- “Metrology Issues in LER and LWR,” Hitachi Technical Forum, San Jose, CA, March 2, 2005.
- “SEM Dimensional Metrology Using a Model-Based Library,” International Workshop on Modeling Electron Transport for Applications in Electron and X-Ray Analysis,” Gaithersburg, MD, November 9, 2004.
- “Issues in Line Edge and Linewidth Roughness Metrology,” International Conference on Characterization and Metrology for ULSI Technology, Richardson, TX, March 16, 2005.

Contributed

- "SEM Modeling for Dimensional Metrology," J. S. Villarrubia, NIST Precision Engineering Division Seminar, Gaithersburg, MD, March 25, 2009.
- "Sensitivity of SEM width measurements to model assumptions," J. S. Villarrubia, SPIE Advanced Lithography, San Jose, CA, February 24, 2009.
- "Collaboration Opportunities in SEM modeling," a presentation to selected faculty of the College of nanoscale Science and Engineering at the Albany Nanotech center, Albany, NY, June 10, 2008.
- "Physics for Secondary Electron Generation," SCANNING, Monterey, CA, April, 12, 2007.
- "High Energy Electron/Solid State Interaction Modeling with MONSEL," Victor Katsap and John Villarrubia, International Vacuum Electronics Conference, Monterey, CA, April 25-27, 2005.
- "Unbiased Estimation of Linewidth Roughness," presented at SPIE conference on Metrology, Inspection, and Process Control, San Jose, CA, March 2, 2005.

Awards:

- Silver medal for development of model-based library method for SEM, awarded by Dept. of Commerce.
- Diana Nyyssonen Metrology Best Paper of 2005 for "Unbiased Estimation of Line-Edge and Line-Width Roughness," awarded by SPIE Advanced Lithography Conference on Metrology, Inspection, and Process Control.

Customers:

- SEMATECH and its member companies
- 5 CD-SEM and other instrument manufacturers
- Users of MONSEL in at least 5 semiconductor manufacturers and 7 universities or research institutes

Collaborators:

- Victor Katsap, Nu Flare Technologies
- Maki Tanaka, Hitachi Ltd.
- Prof. Xiaoping Qian, Illinois Institute of Technology
- Veeco Instruments
- B. D. Bunday, SEMATECH
- Z. -J. Ding, University of Science and Technology of China
- R. Katz, C. D. Chase, R. Kris, and R. Peltinov, Applied Materials

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Photomask Dimensional Metrology

Industry Need:

Significant manufacturing metrology challenges exist beyond scale calibration for the accurate determination of the size width and length of physical features. Measurement of linewidth or critical dimension (CD) continues to be one of the most fundamental dimensional metrology needs in the semiconductor and nanomanufacturing industries.

Semiconductor manufacturers refer to this continually decreasing measurement limit as critical dimension (CD) metrology. The critical dimension size and tolerance decreases as technology progresses. The demand is so complex and ubiquitous that no single metrology technique can provide the entire solution.

Objective:

The goal of this project is to provide industry with accurate and timely dimensional metrology at the nanoscale to enhance U.S. productivity and innovation. In particular, to provide accurate critical dimension metrology for photomask features, and traceable to the meter.

Technical Approach:

Three major techniques are being used within the Photomask Dimensional Metrology Project to mee the objective:

- **Critical-dimension atomic force microscopy (CD-AFM),**
- **Scanning electron microscopy (SEM)**
- **Optical microscopy (OM).**

At this scale, the largest component of measurement uncertainty is usually associated with the interaction of the probe (e.g., mechanical stylus, photons, or a beam of charged particles) with the specimen. The principal challenge in linewidth metrology is to accurately define the position of the physical edge of a feature within the metrology instrument response profile. Each instrument exhibits its own characteristic response profile due to the bandwidth of the electronics, signal collected or probe used. The measurand also contributes to the response as well. The result is an increased linewidth measurement uncertainty. This uncertainty can easily become an undesirable value when dealing with nanometer structures and NIST is continually striving to decrease this measurement limitation.

In order to reduce linewidth measurement uncertainty, new measurement techniques producing sharper edge profiles will be developed and adopted. But, ultimately no method has been able to fully achieve the required resolution, and it becomes necessary to assign the physical edge to a definite position within the broadened signal. This in turn requires understanding and modeling the physical process that produce the broadening so that it can be compensated, and as a result electron beam, scanned probe and optical modeling have become major components of this project.

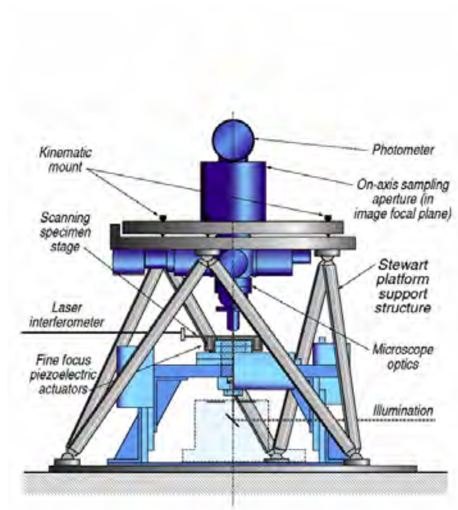

Figure 1. The NIST-designed UV Microscope used for certifying photomasks.

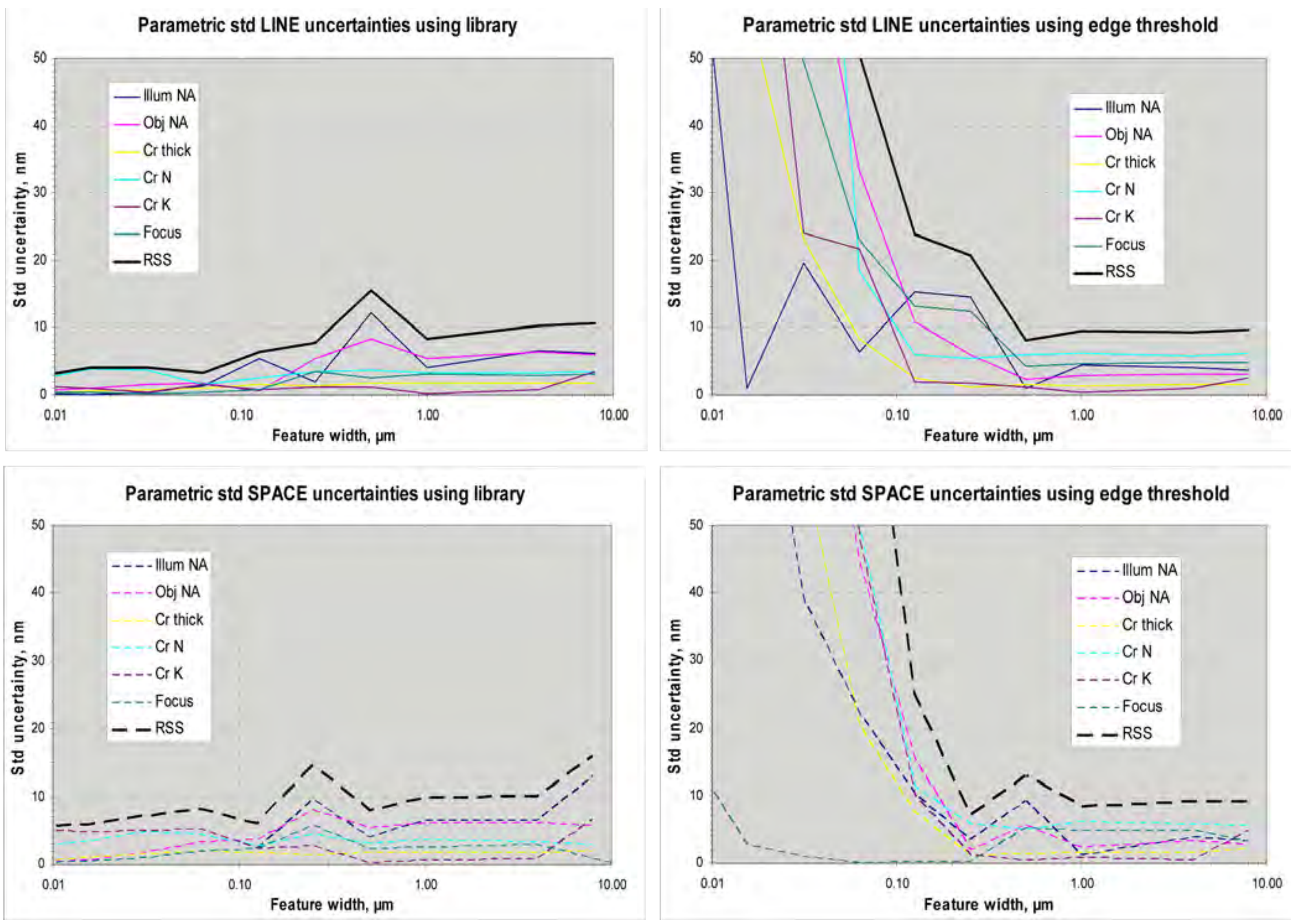

Figure 2. UV Microscope parametric linewidth uncertainties for isolated photomask lines and spaces using Mark Davidson's RCW imaging model, for both threshold and image library dimension extraction methods, using the parameter uncertainties listed in the Table I.

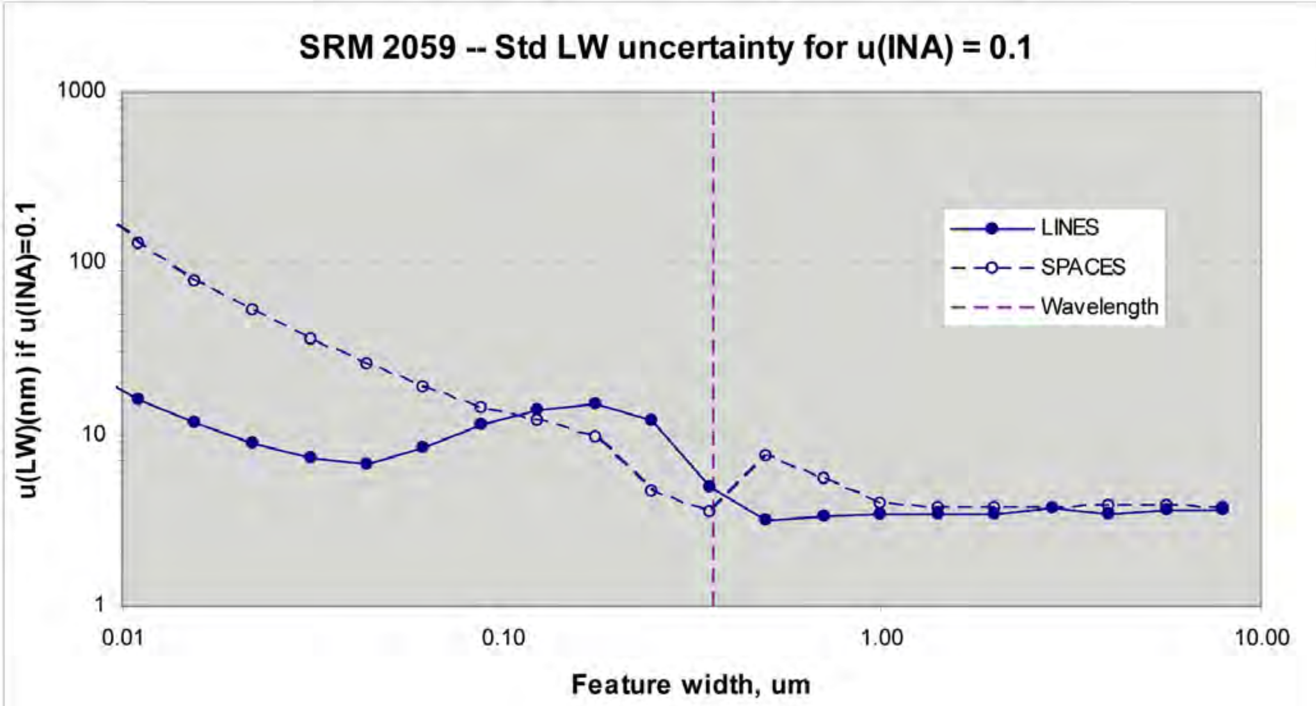

Figure 3. Similar calculation (for Illumination NA only) using Egon Marx's integral equation imaging model and threshold dimension extraction method. Additional parametric uncertainty calculations are underway. (Note the log-log scale).

NIST expertise will focus on improving the effectiveness of the instrument response profile by using shorter-wavelength light in optical microscopy (OM); smaller beam focus spots in scanning electron microscopy (SEM); or sharper, better characterized tips in CD atomic force microscopy (CD-AFM). Such improvements are required to advance this metrology. This contribution to the measurement uncertainty is a greater fraction of the total as feature sizes decrease below 50 nm. Consequently, physics-based modeling is required to understand and overcome this challenge. Validated models for each of the measurement techniques are critical to accurate measurements. In addition, experimental intercomparisons between the various measurement methods are required to further validate the models. To provide traceability, NanoMet will develop custom reference measurement instruments (SEM, SPM and OM) which have been highly engineered to provide traceability through the incorporation of the most accurate laser interferometry. Standard Reference Material (SRM) standards will be certified with these reference measurement instruments to accurately calibrate production instrumentation.

Project Overview:

The ideal technique for photomask linewidth measurements is the transmission optical UV microscope because optical transmission imaging produces relatively simple high contrast images with a well-defined baseline. Also, transmission optical microscopy emulates the way the photomask is used during the wafer exposure process, thus enhancing the effectiveness of the standard.

The NIST scanning UV microscope (shown in Figure 1) uses on-axis sampling to reduce aberration and distortion effects. The position of the scanning stage is measured by a laser interferometer. NIST will develop accurate physics-based modeling to deduce the object dimensions from the microscope image. Image modeling will also extend the limits of optical metrology to feature sizes well below the wavelength of light used, as demonstrated by Dianna Nyyssonen at NIST in the 1980s when critical dimensions were micrometer in size. The challenge is to measure state of the art chromium photomasks and phase shifting masks with sub-0.25 micrometer structures. NIST will also work on a next generation photomask standard. This standard will likely contain, in addition to isolated lines, spaces, and pitch patterns from ≈100 nm wide, a variable set line/ space arrays as well as large scale 2-dimensional features in

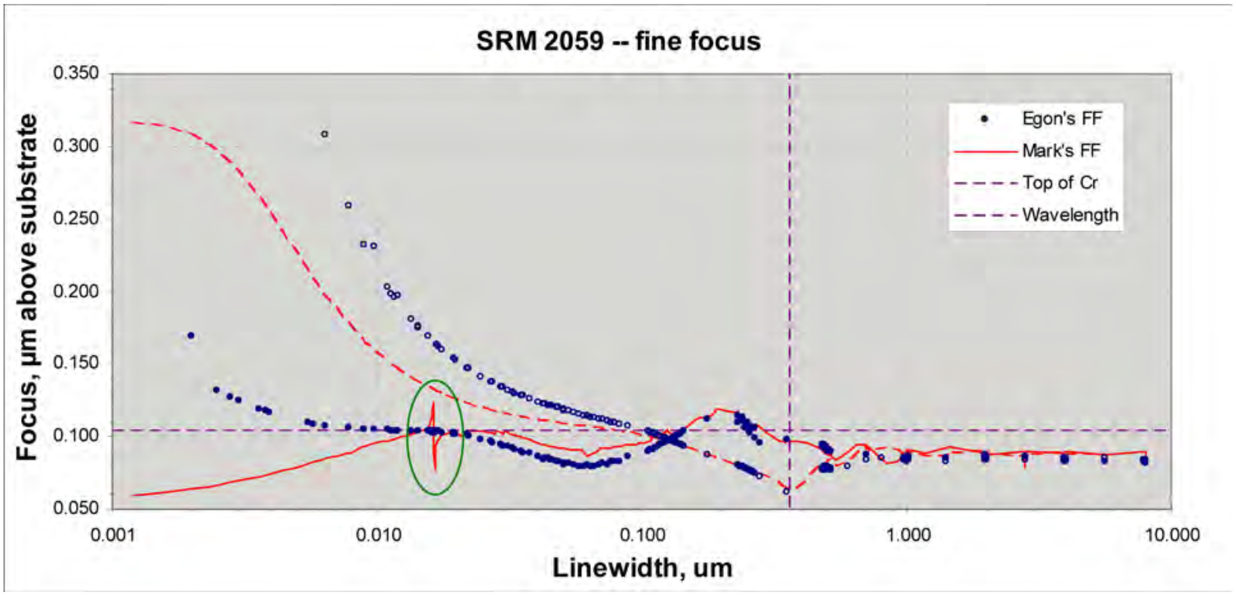

Figure 4a. Model results for best focus of isolated photomask features. Open circles and dashed lines are space features, solid circles and lines are line features.

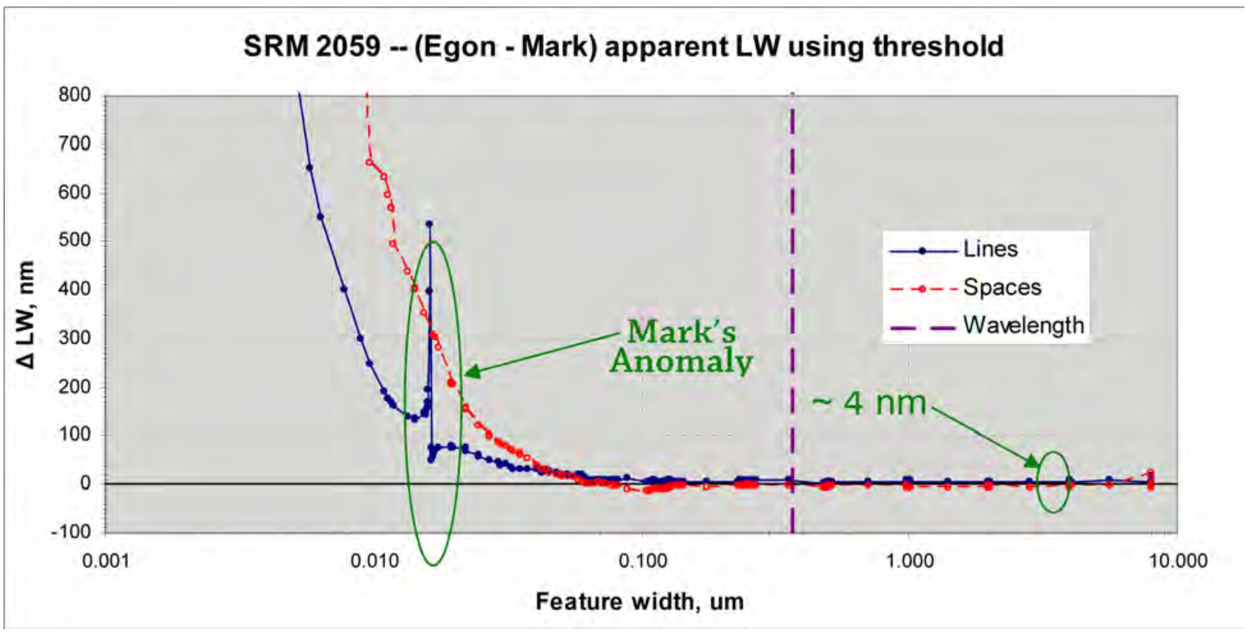

Figure 4b.Apparent feature width difference between the integral equation and RCW models (using threshold dimension extraction method) is about 6 nm for features larger than 100 nm, but increases for smaller features.

response to requests from the machine vision industry. This project will also develop an essential suite of techniques to calibrate and optimize the optical system errors. This new area of research is critical to improving model to experiment agreement in model-based linewidth measurements.

Experience with a model developed by Mark Davidson (Spectel Company) implies the uncertainty explosion seen here for small features will not appear when the image library (now being constructed) feature dimension extraction method is used. All features are at best focus; see below. Our optical image modeling research to date supports the conclusion that there is no physical lower limit to the isolated feature size on a photomask or wafer which can be measured optically (unlike dense feature arrays), regardless of the wavelength (Fig. 2). No exotic microscope designs are required; the ultimate limiting factor is the signal-to-noise ratio.

Table I.
Parameter uncertanites used for linewidth uncertainty correlations

Parameter	Cr Thickness	Cr N	Cr K	Illum. NA	Obj NA	Defocus
Nominal	0.1035 µm	1.843	-2.195	0.6	0.9	0
Uncertainty	0.003 µm	0.5	0.5	0.1	0.05	0.1 µm

Example of best focus for photomasks:

There are several ways to define best focus in a microscope, but in our UV Microscope best focus is defined as that focus position which gives the maximum image acutance at the 50% image intensity point. This definition is realizable in the laboratory and is unambiguous for a reasonable range of through-focus images. While Mark Davidson's RCW imaging model and Egon Marx's integral equation model are in pretty good agreement for features wider than 100 nm, they diverge in best focus and apparent feature size for smaller features, as shown below.

....transmission optical microscopy emulates the way the photomask is used during the wafer exposure process, thus enhancing the effectiveness of the standard.

Again, experience with Mark's model implies the divergence of apparent linewidths seen here for small features will mitigate when the image library feature dimension extraction method is used (except for Mark's anomaly at 16.5 nm lines, seen also in the focus graph). The divergence in best focus is unexplained; feature dimension extraction is not involved.

Accomplishments:

- Collaborated directly with representatives of the photomask industry and the SEMATECH Mask Advisory Steering Council to determine their requirements in the next generation NIST photomask linewidth standard.
- Completed the assembly and recalibration of the optical transmission photomask instrument. Characterize all optical components to ensure optimum instrument performance.
- Completed a bilateral intercomparison on photomask linewidth standards between NIST and Physicalishe Technicshe Bundesanstalt (PTB) to ensure agreement between the leading two international suppliers of photomask calibration standards.
- Improved the optical image modeling capability to better agree with actual AFM images for confirmation of optical measurements.
- Incorporated the new specimen stage into the UV Microscope.
- Completed Nano1, the BIPM international intercomparison on photomask linewidth standards that will be piloted by NIST, to ensure measurement agreement between those international standards labs engaged in photomask linewidth metrology.
- Continued annual presentations of the short course Nanoscale metrology--theory and practice (J.Potzick and B.Grenon) at the annual SPIE/BACUS Photomask technology Symposium.

Publications:

2009

- Quintanilha, Sohn, Howard, Stocker, Silver, Potzick, "Photomask metrology using a 193-nm scatterfield microscope," SPIE/BACUS Photomask Technology Symposium, 7488-55 (2009)
- Bodermann, Bergmann, Buhr, Häßler-Grohne, Bosse, Potzick, Dixson, Quintanilha, Stocker, Vladar, Orji, "Results of an international photomask linewidth comparison between NIST and PTB," SPIE/BACUS Photomask Technology Symposium, 7488-51 (2009)
- Smith, Tsiamis, McCallum, Hourd, Stevenson, Walton, Dixson, Allen, Potzick, Cresswell, Orji, "Comparison of Measurement Techniques for Linewidth Metrology on Advanced Photomasks," IEEE Transactions on Semiconductor Manufacturing, 22, 1, pp. 72-79 (2009)
- Marx E., Potzick, "Computational Parameters in

Simulation of Microscope Images", Progress In Electromagnetics Research Symposium, Beijing, China (2009)

- Dixson R., Potzick, Orji, "Re-Calibration Of The SRM 2059 Master Standard Using Traceable Atomic Force Microscope Dimensional Metrology," Frontiers of Characterization and Metrology for Nanoelectronics, Albany, NY (2009)
- Potzick J. "Image library approach to evaluating parametric uncertainty in metrology of isolated feature width," Proceedings of SPIE Metrology, Inspection, and Process Control for Microlithography XXIII, San Jose, Calif. (2009)

2008

- Potzick J, Dixson, Quintanilha, Stocker, Vladar, Buhr, Häßler-Grohne, Bodermann, Frase, Bosse, International photomask linewidth comparison by NIST and PTB," Procedings SPIE Photomask (BACUS), 7122-97 (2008)
- Potzick, J. "Accuracy of Optical Dimensional Metrology at the Nano-scale", Microscopy & Microanalysis 2008 Meeting, Albuquerque, New Mexico (2008)
- Smith J., Tsiamis, McCallum, Hourd, Stevenson, Walton, Dixson, Allen, Potzick, Cresswell, Orji, "Comparison of Measurement Techniques for Advanced Photomask Metrology", International Conference on Microelectronic Test Structures, Edinburgh, Scotland (24-27th March, 2008)
- Potzick, J. "Limits of optical dimensional metrology at the nano-scale," 2nd Tri-National Workshop on Standards for Nanotechnology, Gaithersburg, Md. USA (Feb. 6, 2008). (invited)
- Potzick, J. Grenon, Nanoscale metrology--theory and practice, Course Notes, presented at SPIE BACUS 2007, BACUS 2008

2007

- Dixson R., Orji, Potzick, Fu, Cresswell, Allen, Smith, Walton, "Photomask Applications of Traceable Atomic Force Microscope Dimensional Metrology at NIST," SPIE Photomask Technology 27th Annual Symposium, vol. 6730-117 (2007).
- Potzick, J., "Linewidth Standards and CD Metrology," Workshop:Critical Dimension Standards: The Past, Present, and Future, SPIE Symposium on Advanced Lithography (2007) (invited)
- Potzick, J., Marx, Davidson, "Accuracy in Optical Image Modeling," Proceedings of SPIE Metrology, Inspection, and Process Control Conference for Microlithography XXI, vol. 6518-39 (2007).
- Attota R.,, Silver, Potzick, "Optical Illumination and Critical Dimension Analysis Using the Through-Focus Focus Metric Method," SPIE Optics and Photonics Conference (2007)

2006

- Potzick, J., Marx, Davidson, "Parametric Uncertainty in Optical Image Modeling," Proceedings of BACUS Symposium on Photomask Technology, vol. 6349-187 (2006).

2005

- Potzick,J. "A benefit/cost model for metrology in manufacturing," Proc. ISMI Symposium on Manufacturing Effectiveness, SEMATECH, (2005)
- Marx E., Potzick, "Simulation of optical microscope images for photomask feature size measurements," 2005 Digest of the IEEE Antennas and Propagation Society International Symposium, 2116-9 Vol. 3B pp. 243-246 (2005).
- SEMI Standard P35-0704, Terminology for Microlithography Metrology, SEMI International Standards, 3081 Zanker Rd., San Jose, California 95134
- Potzick, J., "Photomask feature metrology," Ch. 21 of A Handbook on Mask Making Technology, Syed A. Rizvi, Ed., Marcel Dekker Inc. New York (2005). (invited)

Customers:

- Photronics
- Toppan Photomasks, Inc.

Collaborators:

- SEMATECH
- VLSI Standards
- IBM
- Intel
- SEMI International Standards
- Physikalisch Technische Bundesanstalt (Germany)
- BIPM (France)

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Overlay Instrument
and
Wafer Target Designs

Industry need:

This project develops measurement techniques and provides standards to enable accurate overlay and registration metrology with subnanometer accuracy for manufacturing and process control of the most advanced, fastest semiconductor devices.

A significant challenge for the semiconductor manufacturing industry is to develop advanced metrology techniques for overlay and registration to enable the continued long-term advance of device performance based on the stringent International Technology Roadmap for Semiconductors (ITRS) industry guidelines. As the technology advances and critical dimensions shrink, the requirements for overlay tolerance and accuracy become more stringent. Currently, optical techniques are most widely used for this type of metrology as the semiconductor industry needs economical and high throughput overlay measurement techniques. Important attributes of the complete overlay measurement process include the overlay target design and the measurement methodology, each one having its own set of current and future semiconductor industrial needs.

The metrology target should emulate the device dimensions to eliminate or reduce errors due to dimensional mismatch between actual devices and metrology targets in the lithography and fabrication process. The overall size of the target should also be made as small as possible to reduce the valuable real estate needed for non-value added targets. Additionally, conventional image-based overlay targets occupy large areas, which is in contradiction with the increasing need to place the overlay targets in the active area, as opposed to in the scribe-line area. The need stems from the measurement errors that result from overlay measurements made in different locations. Overlay measurement using targets placed in the active area have been found to more closely indicate the actual device overlay offset and are therefore more accurate. Reduction of overlay target dimensions saves precious real estate on the wafer and results in improved yield and a reduction in the overall manufacturing cost of computer chips.

The recent advance of double patterning techniques, which directly couple overlay and line measurements resulting in reduced critical feature dimensions, has made this metrology still more urgent. Double patterning overlay metrology is considered much more challenging as it requires considerably higher accuracy. This lithography technique is also likely to require different types of overlay targets or measurement methods as all of the features are in the same level as opposed to different levels seen in traditional overlay measurements.

Conventionally, image based overlay targets have been widely used. However, they occupy large areas and are susceptible to proximity effects and large feature edge bias that further diminish overlay measurement accuracy when the target size is substantially reduced. Therefore, there has been a recent need and push for new types of overlay measurement processes that use optical signal scattering rather than image-based analysis while still providing the required measurement throughput.

Project Objective:

The overlay metrology project is an internationally recognized effort with the goal of developing techniques and targets for improved overlay metrology, primarily for the semiconductor industry. A primary objective is to develop advanced position metrology, techniques and standards for overlay and registration that meet the critical semiconductor industry needs and those future challenges of the demanding nanomanufacturing industry. The relative overlay of features from different manufacturing process levels is considered one of the most demanding measurement requirements due to the direct effects on device performance. In response, NIST has been working closely with industry leaders to develop the infrastructural metrology to ensure the accurate placement (registration) of multiple layers with sub-nanometer accuracy.

Since optical techniques are often best suited to these tasks, research into novel optical overlay and registration instrumentation and target structures is essential. In semiconductor applications, development of new instrument innovations and target designs are jointly pursued with the leading industrial consortium, SEMATECH and other leading edge semiconductor manufacturers. New metrology target design goals enable improved overlay resolution in a smaller, in-chip format composed of sub-resolution features. It is critical to develop high-resolution optical overlay techniques that are extensible for several manufacturing generations. The progression of technology demands that new generations of SRMs meet new production challenges and must continually be developed to ensure the standards and measurement needs are met ensuring continued progress of the industry.

SRMs for overlay calibration are designed and fabricated jointly with the industry to ensure industry challenges are correctly met. The collaborative implementation of new target designs, instrument optimization and modeling, and calibration techniques includes new reticle design and wafer fabrication in the continuing effort undertaken jointly with key industrial partners. The project has relied on close collaboration with industry leaders in optical tool development and users of overlay metrology tool sets as well as with SEMATECH. NIST continues to work with the industrial partners to gain access to state of the art lithography processes for wafer and target fabrication ensuring the progress of the industry.

Technical Approach:

An essential element of this project is the identification of current industrial needs and a continued verification of technical goals that meet these changing technology needs. Determination of the need is done in close collaboration with the semiconductor industry and SEMATECH. The technical goals for overlay target design and measurement method research is implemented directly based on the need.

Extensive electromagnetic scattering and optical modeling is performed on new target designs and modeling methods are used to develop a thorough understanding of all the variables and parameters that affect overlay measurements including optimization of the parameters. Two commercially available optical simulation models (rigorous coupled wave guide analysis (RCWA) and finite difference time domain (FDTD)) and two in-house developed models (RCWA and integral equation method (IEM)) are in use for this project. An additional high speed three-dimensional model based on the finite difference time domain (FDTD) method is being developed in-house. Simulation results from different models are routinely compared for accuracy and consistency.

Implementation of new overlay methods often requires construction of new optical tools or extensive modification of existing tools. One dedicated existing optical tool has been extensively modified to meet our overlay measurement and calibration needs. A second Scatterfield optical tool was designed and developed in-house. A third fully custom, state-of-the-art metrology microscope that operates at 193 nm wavelength has also been designed and fabricated in-

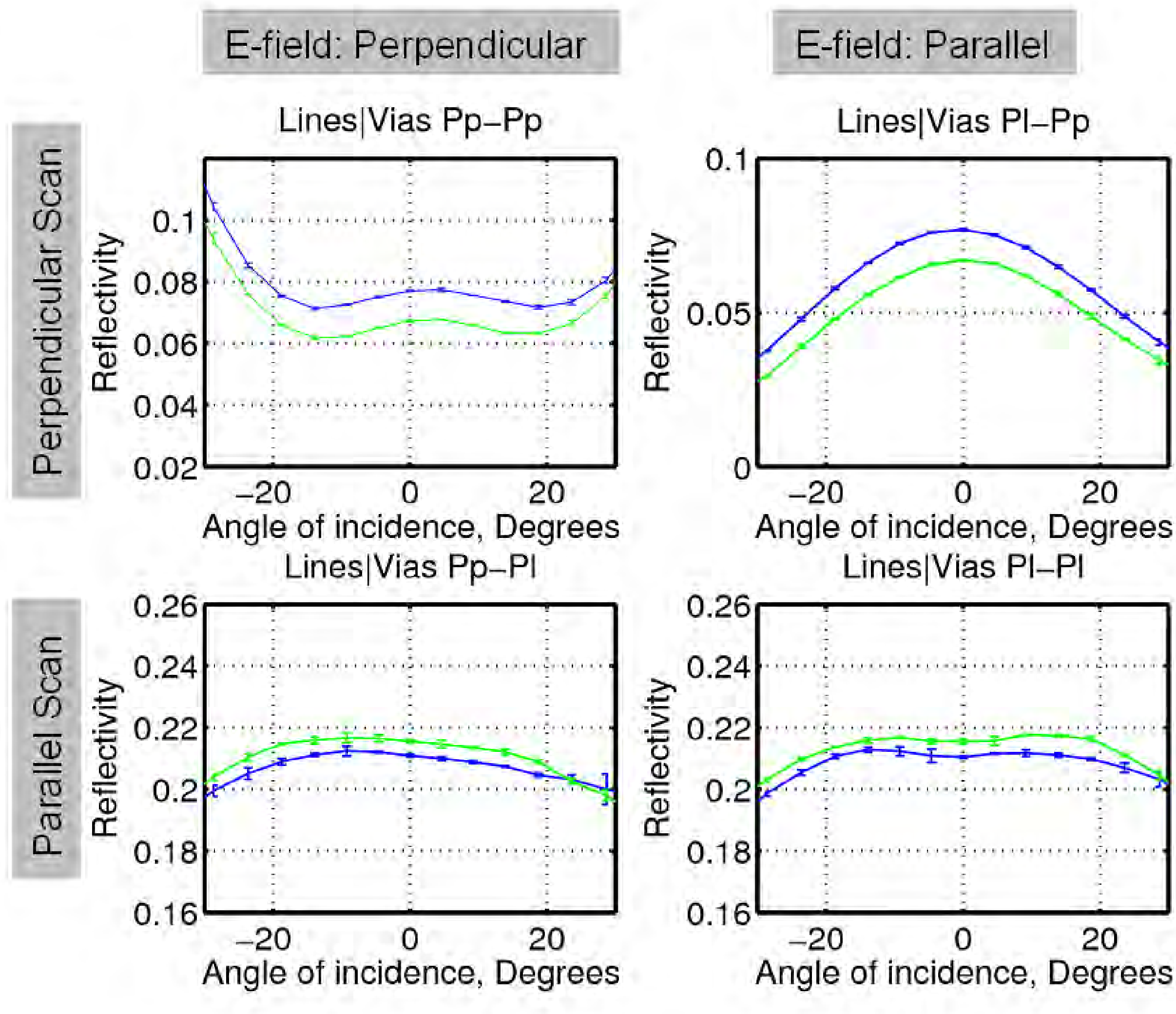

Figure 1. Scatterfield overlay measurements showing different polarizations and scan directions for complex via overlay structures.

house for various applications, including defect and overlay imaging.

New optimized targets are fabricated in both the NIST NanoFab or acquired from SEMATECH to test and validate models and implement quantitative optical measurement procedures with sub-nanometer sensitivity and accuracy using the above mentioned optical tools. In our extensive collaboration with SEMATECH, a series of next generation Overlay Metrology Advisory Group (OMAG) overlay test targets have been designed and developed in response to changing industrial needs. The overlay metrology project has been closely involved since the inception of the OMAG reticle design sets including more recent generations of the OMAG3, OMAG4 and AMAG5 overlay target test wafers.

Scatterfield microscopy is one of the methods developed in this project for advanced optical nanometrology based on optical signal analysis rather than optical image processing. This technique has potential applications for overlay measurements. Changes in the reflectance of an overlay target occurring as the illumination angle of incidence is scanned can be used to evaluate overlay. An example of 25 nm overlay measured for vias is shown in Fig. 1. Following technique initial positive results further study is now underway to fully develop an understanding of this technique for overlay measurements.

Through-focus scanning optical microscope (TSOM) is another new optical method developed here for nanometrology. A conventional optical microscope is used to collect the images of a given target as it is scanned along the focus axis. Two dimensional TSOM images generated by processing acquired optical images show high sensitivity to nanometer level changes in various dimensions of the target. A new set of overlay targets designed and tested using this method showed good sensitivity to overlay as shown in Fig. 2. The TSOM based overlay targets are well

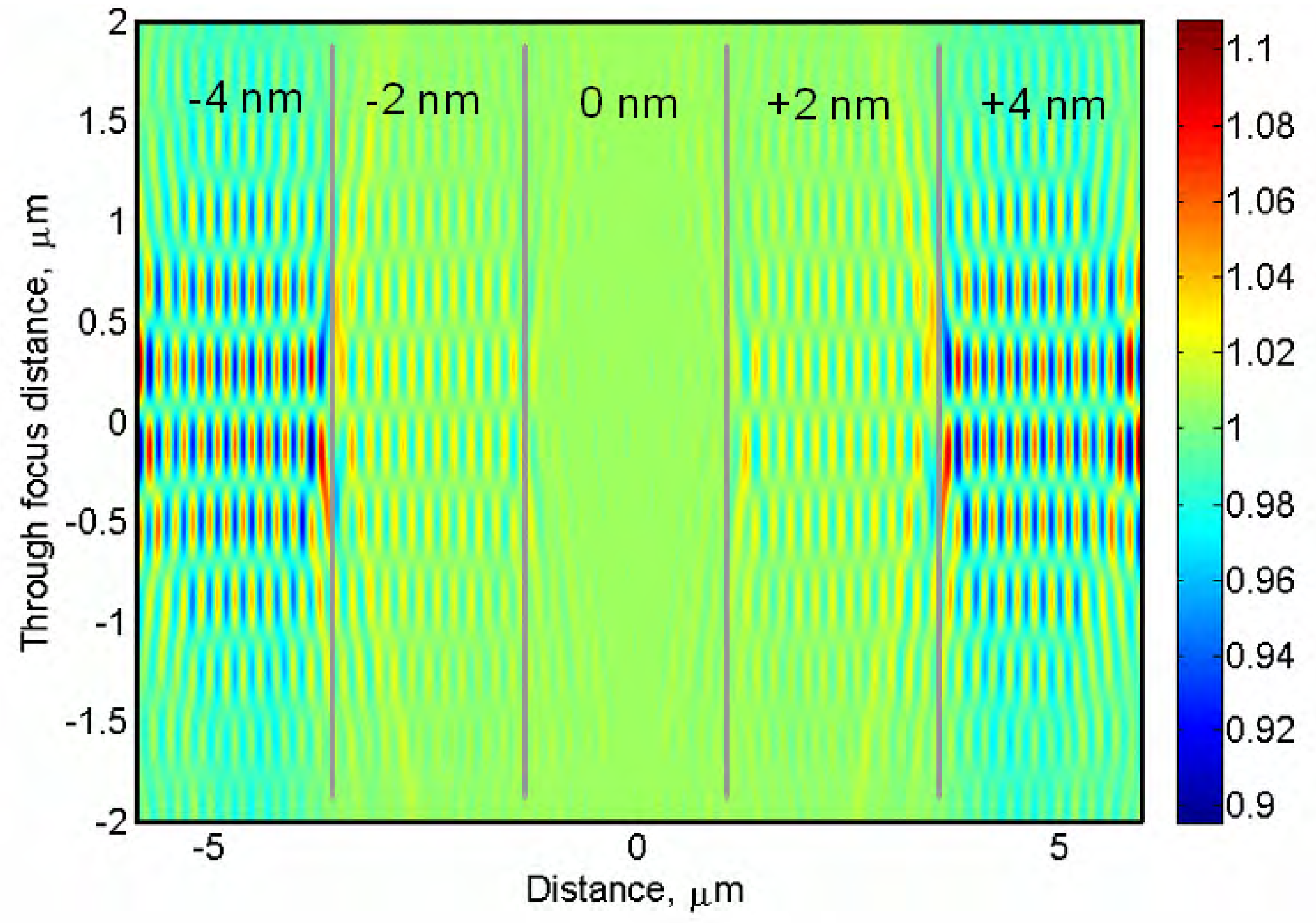

Figure 2. TSOM image of a composite overlay target designed for double patterning showing sensitivity for different overlay values indicated.

suited for overlay requirements for double patterning.

Impact/Benefits:

One major impact of the overlay project was the incorporation of NIST-developed methods into the hardware tool sets of leading metrology tool manufacturers. Target reversal procedures developed in the Nanomanufacturing program have been installed that help achieve the lowest tool induced shift (TIS) - a measure of tool accuracy for overlay measurement, location in the field of view.

The Scatterfield optical platform developed at PED has been adopted by several companies and research centers. The overlay metrology methodology and platform has been tested, validated and implemented in optical research instrumentation and recently in production metrology hardware.

The overlay standard reference material 5000 (SRM 5000) developed at PED has been used by leading industry users to calibrate their optical overlay tools. This work was awarded a Silver medal from DOC.

Two-dimensional grids have been fully calibrated using the Nikon 5i and linescale interferometer. These measurements made in combination with leading edge industry tools resulted in world class measurement uncertainties.

Technical project leads have contributed extensively to the SEMI standards committee and have chaired the overlay metrology subcommittee and co-chaired the main SEMI Microlithography Patterning Committee.

Accomplishments:

- Industry adaptation of a number of overlay metrology techniques and hardware configurations based on NIST-published material.

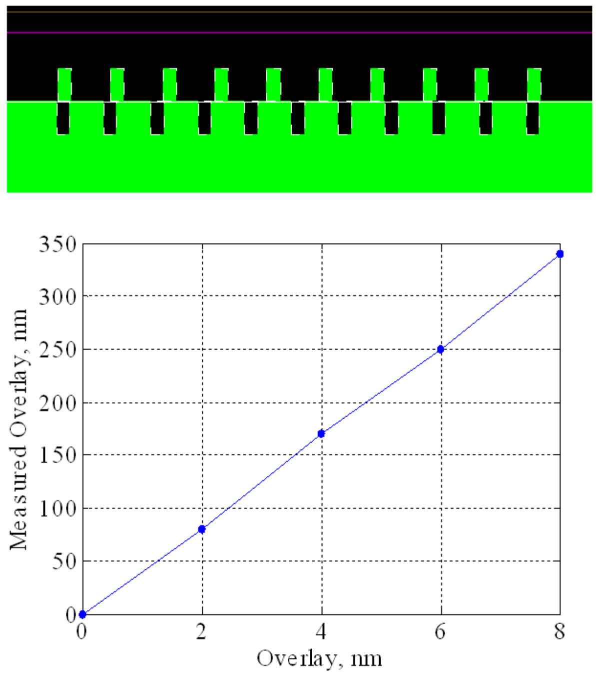

Figure 3. An example of a supertarget design. This new patented design allows conventional imaging hardware to be used with dense sub-wavelength features.

> **"A significant challenge for the semiconductor manufacturing industry is to develop advanced metrology techniques for overlay and registration to enable the continued long-term advance of device performance based on the stringent International Technology Roadmap for Semiconductors" [ITRS Guidelines]**

- Vertical and horizontal proximity studies have received recent attention as they have uncovered key attributes of scatterometry for overlay complications.
- SEMATECH applied for a joint NIST/SEMATECH patent in Feb, 2008, for the new super-resolution overlay target (Figure 3). These targets have the potential to change the target designs and methodology widely in used by the industry.
- In the continuing collaboration between the NIST advanced scatterfield optical research and SEMATECH, Rick Silver has been invited to give numerous presentations to the Advanced Metrology Advisory Group (AMAG) and the Defect Metrology Advisory Group (DMAG) throughout the previous 5 years.
- TSOM based overlay.
- Final report submitted to SEMATECH on the contract to investigate advanced optical overlay metrology including a gauge study of scatterfield microscopy applications and dipole illumination evaluation.
- Several Patent applications and invention disclosures.
- Authored a book chapter on EUV Metrology. This is a new book to be published by McGraw Hill and will be the benchmark publication for semiconductor manufacturing challenges using next generation EUV lithography
- Overlay using 193 nm tool
- New techniques for overlay metrology in double patterning
- Invited presentation at the Albany Center for nanoscale Research titled "High-resolution Optical Metrology
- In-chip targets have been extensively published and developed at NIST and have been used in several industrial studies and applications.
- Redundant-edge overlay targets were initially developed in a joint effort between this NIST project and SEMATECH overlay metrology advisory group. These target designs have found their way into a number of current overlay targets using redundant edge techniques.
- 2-D grids for registration calibration were developed into SRM 5001. Several of these have been sold and are used for calibration of photomask registration.

- Richard Silver was elected Fellow of the SPIE and received recognition for the achievement at the 2008 Advanced Lithography symposium in large part due to work in overlay metrology.
- Reference metrology for overlay was developed at SEMATECH using the NIST calibrated SRM 5000. This calibrated overlay standard has been used by several companies for the calibration of production metrology hardware.

Publications:

2009

- Attota, R., Germer, T., and Silver, R.M., 2009. Nanoscale measurements with a through-focus scanning optical microscope. Future Fab, 30, p. 83-88.
- Attota, R., Stocker, M., Silver, R.M., Heckert, A., Zhou, H., Kasica, R., Chen, L., Dixson, R., Orji, G., Barnes, B., Lipscomb, P., 2009. Through-focus scanning and scatterfield optical methods for advanced overlay target analysis. Proc. SPIE, 7272, p. 727214.
- Silver, R.M., Zhang, N., Barnes, B., Zhou, H., N. Heckert, Dixson, R., Germer, T., and Bunday, B., 2009. Improving Optical Measurement Accuracy using Multi Technique Nested Uncertainties. Proc. SPIE, Vol. 7272, p. 1.

2008

- Attota, R., Germer, T., and Silver, R.M., 2008. Through Focus scanning optical microscope imaging method for nanoscale dimensional analysis. Optics Letters 33, p. 1990-1992.
- Attota, R., Silver, R.M., and Barnes, B., 2008. Optical Through Focus Technique that Differentiates Small Changes in Line width, Line Height and Sidewall Angle for CD, Overlay and Defect Metrology Applications. Proc. SPIE, Vol. 6922, p. 69220E.
- Attota, R., Silver, R.M., Dixson, R., 2008. Linewidth measurement technique using through-focus optical images. Applied Optics 47, p. 495-503.
- Silver, R.M., Barnes, B., Heckert, A., Attota, R., Dixson, R., and Jun, J., 2008. Angle resolved Optical Metrology. Proc. SPIE, Vol. 6922, p. 69221M.
- Patrick, H., Attota, R., Barnes, B., Germer, T., Dixson, R., Stocker, M., Silver, R.M., and Bishop, M., 2008. Optical critical dimension measurement of silicon grating targets using back focal plane scatterfield microscopy. J. Micro/Nanolith. MEMS MOEMS 7, 013012.

2007

- Silver, R.M., Barnes, B., Attota, R., Jun, J., Stocker, M., Marx, E., and Patrick, H., 2007. Scatterfield Microscopy to Extend the Limits of Image-based Optical Metrology. Applied Optics, Vol. 46, p. 4248-4257.
- Silver, R.M., Germer, T., Attota, R., Barnes, B., Bunday, B., Allgair, J., Marx, E., and Jun, J., 2007. Fundamental Limits of Optical Critical Dimension Metrology: A Simulation Study. Proc. SPIE, Vol. 6518, p. 6518OU.
- Sohn, Y.J., and Silver, R.M., 2007. Kohler Illumination Analysis for High Resolution Optical Metrology using 193 nm Light. Proc. SPIE, Vol. 6518, p. 65184V.
- Silver, R.M. Attota, R., Barnes, B., and Zhou, H., 2007. Theoretical Evaluation of Scatterometry for Overlay Applications. SEMATECH Tech. Transfer Doc.
- Barnes, B.M., Howard, L.P., Jun, J., Lipscomb, P., and Silver, R.M., 2007. Zero-order imaging of device-sized overlay targets using scatterfield microscopy. Proc. SPIE, 6518, 65180F.
- Silver, R.M., Attota, R., and Marx, E., 2007. Model-based Analysis of the Limits of Optical Metrology with Experimental Comparisons. Proc. SPIE, Vol. 6617, p. 6617OW.
- Stocker, M.T., Silver, R.M., Attota, R., Jun, J., 2007. Calibrated Overlay Wafer Standard. NIST Special Publication SP-260-165, p. 260-165.

2006

- Attota, R., Silver, R.M., and Potzick, J., 2006. Optical Illumination and Critical Dimension Analysis Using the Through-focus Focus Metric Method. Proc. SPIE, Vol. 6289, p.62890Q-1.
- Attota, R., Silver, R.M., Bishop, M.R., and Dixson, R.G. 2006. Optical critical dimension measurement and illumination analysis using the through-focus focus metric. Proc. SPIE, 6152, 61520K.
- Silver, R.M., Barnes, B., Attota, R., Jun, J., Filliben, J., Soto, J., Stocker, M., Lipscomb, P., Marx, E., Patrick, H., Dixson, R., and Larrabee, R., 2006. The Limits of Image-based Optical Metrology. Proc. SPIE, Vol. 6152, 61520Z.
- Lipscomb, P., Allgair, J., Bunday, B., Bishop, M., Silver, R.M., Attota, R., and Stocker, M., 2006. Calibration Optical Overlay Measurements. Proc. SPIE, Vol. 6152, 615211.

- Kandel, D.K., Adel, M., Frommer, A., Levinski, V., Rapoport, A., and Silver, R.M., 2006. Imaging Simulations of Optimized Overlay Marks with Deep Sub-resolution Features. Proc. SPIE, Vol. 6152, 61522X.
- Barnes, B., Howard, L., and Silver, R.M., 2006. Illumination Optimization for Optical Semiconductor Metrology. Proc. SPIE, Vol. 6289, p. 6289OP.

2005

- Attota, R., Silver, R.M., Germer, T., Bishop, M., Larrabee, R., Stocker, M., and Howard, L., 2005. Application of Through-focus Focus-metric Analysis in High Resolution Optical Microscopy. Proc. SPIE, Vol. 5752, p. 1441-1449.
- Silver, R.M., Attota, R., Stocker, M., Bishop, M., Howard, L., Germer, T., Marx, E., Davidson, M., and Larrabee, R., 2005. High-resolution Optical Metrology. Proc. SPIE, Vol. 5752 p. 67.

Customers

- SEMATECH, ISMI
- IBM
- Intel
- KLA Tencor
- Nanometrics

Collaborators

- KLA Tencor
- Nanometrics
- Nova
- AMD
- SEMATECH, ISMI
- IBM
- Intel
- SEMI International Standards

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Next-Generation Nanometrology Program- *Solving Industry Needs of Tomorrow*

Challenge

Develop measurement science and technology approaches to address the measurement challenges of next-generation nanofabrication, nanotechnology and nanomanufacturing.

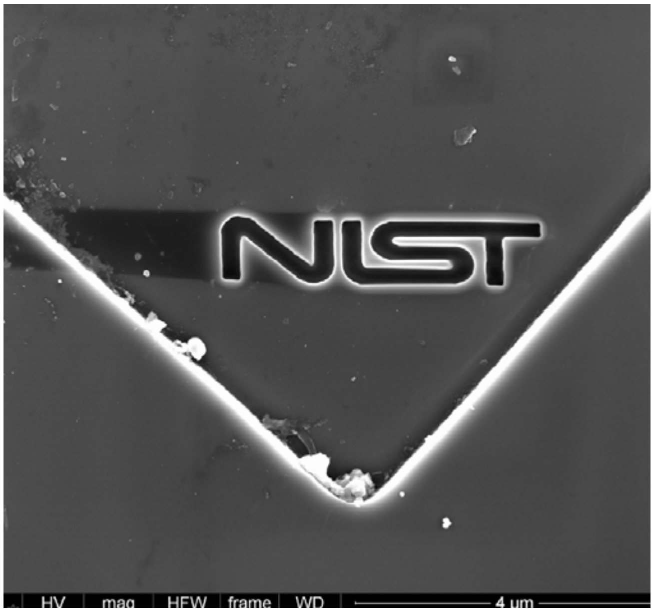
Overview

Meeting the electronics industry's rallying cry for ever smaller and faster components requires more than improvements in manufacturing techniques: engineers must be able to see and measure what they are building. The limits of conventional imaging have been reached and passed, so NIST is devising new ways to measure and image nanoscale components quickly and accurately. By pushing optical microscopy beyond its traditional limits, improving the speed of non-optical techniques, developing methods for the accurate measurement of nanoparticles (crucial for advanced medicine delivery, among others), determining new calibration standards, and developing accurate methods for measuring 3-D features, this program addresses the measurement challenges of next-generation fabrication.

Program Description

The next generation of nanotechnology manufacturing will hit a wall without new measurement techniques to image, analyze, and refine the atomically dimensioned devices now being envisioned. Already, scales are so small that fabrication is beginning to rely on self-assembly, bio-manufacturing, and other methods that require no direct human intervention. Manufacturing on still smaller scales requires a complete overhaul of measurement science—merely improving current methods will not be enough to meet the challenges. Few companies can spend the necessary time and money brainstorming to creatively meet such long term needs, so NIST, with its expertise in measurement techniques and commitment to providing a competitive edge to U.S. industry as a whole, is addressing the challenge with its Next-Generation Nanometrology Program (NextGen).

NextGen has several strategies to develop methods to accurately measure nanoscale features. Current measurement techniques are not only slow—scanning a sample by sending an electron beam back and forth across it, for example—but often entail damage or destruction of a product to see its interior. Current optical microscopes can rapidly examine objects without doing



damage, but can only measure features down to about the wavelength of light. NextGen will develop advanced optical microscopes using a technique called scatterfield microscopy, which can extract quantitative information from measurements of the angle, polarization and other attributes of light bouncing off a sample. While this technique does not create images, it can reveal the shape of an object at a scale less than 1/20th the wavelength of light—close to atomic dimensions. The grand challenge will be to do this for randomly distributed particles, as opposed to ordered crystals and semiconductors. This would allow determination of the size and distribution of 5 nm platinum particles attached to 100 nm carbon particles scattered throughout a fuel cell, for example.

NextGen also aims to improve non-optical microscopy. The flagship is the scanning helium ion microscope, a novel instrument related to the scanning electron microscope that scans with a beam of helium ions instead of electrons. The technique has better resolution than scanning electron microscopy, and can also modify a sample by implanting helium ions or milling away material at the surface to reveal additional features.

NextGen also focuses on measuring nanoparticles, a challenge that is vital to U.S. industry because of nanoparticles' promise for the pharmaceutical industry along with their possible environmental risks. NIST is also investigating the "fate of nanoparticles in biological systems" thus developing the accurate dimensional metrology for the environment, health and safety. In addition, NIST is working with Georgetown University Medical Center on studies of 200 nm phospholipid particles that are used to deliver targeted medicines or contrast agents for MRIs and X-rays. In neither capacity do these particles yet perform consistently. But it is difficult to analyze their structure with microscopes since their shape deforms when they bind to a surface, such as a microscope slide. NIST will develop better imaging methods and will creatively work around related problems, perhaps by fine-tuning the stickiness of the slide or by learning how to infer the nanoparticle's true properties despite the deformation.
The final NextGen focus represents a jump from NIST's history of creating exact measurement standards to distribute for calibration purposes. Researchers wish instead to provide measurement methods that allow companies to create their own standards—such as by publishing a reference value for some natural standard, like a crystal lattice spacing. Customers could then prepare their own crystal as a calibration standard.

Projects

The Next-Generation Metrology Program meets its objectives through 5 projects

- Scanning Particle-Beam Microscope Innovations
- Atomic Force Microscope (AFM) Nanoparticle Metrology
- Nanoparticle Manipulation Metrology
- Atom-Based Dimensional Metrology
- Molecular Measuring Machine
- High-Throughput Nanometrology with Scatterfield Microscopy

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Scanning Particle-Beam Microscope Innovations

Industry Need:

Nanotechnology is already facing dimensional measurement and characterization challenges that far exceed the capabilities of current measurement science and techniques. Three-dimensional nanometer-scale objects and features must be measured with unprecedented precision and uncertainty, and with high throughput. New dimensional measurement technologies and standards must be conceived and developed to meet the measurement challenges of the emerging U.S. nanotechnology industry.

This project is about the development of future nanometer-scale dimensional measurement methods and techniques that will serve various research and industrial use of scanning electron and ion microscopes.

Project Objective:

It is the goal of the Project to develop accurate methods for the characterization and three-dimensional metrology of nanoscale materials and features. A significant metrology challenge for next-generation manufacturing is the need to identify and measure true three-dimensional (3D) features embedded in complex matrices. The Project will develop advanced scanned particle beam metrologies to address this need either through high accelerating voltage penetration-imaging of relatively thin slices of the sample with a scanning electron microscope, or through ion-milling into the sample to expose the features of interest for subsequent examination with dual column electron/ion microscopes. In addition, the Project will advance the development of a scanning helium ion microscope (HIM) for high-resolution metrology, characterization and high-throughput nano-scale materials production. The HIM is a new and revolutionary instrument with resolution and depth-of-field far exceeding current scanning electron microscopes, thus providing enhanced 3D surface characterization and metrology.

Project Overview:

Scanning electron microscopy (SEM) is one of the workhorse techniques for imaging and characterization for nanotechnology. The Scanning Particle-Beam Microscope Innovations Project is now developing various advanced methods that enhance the SEM instrument's capabilities and new metrology methods.

Nanotechnology is already facing dimensional measurement and characterization challenges that far exceed the capabilities of current measurement science and techniques.

Advanced, High-performance Sample Stages.

NIST is developing high-performancesample stages equipped with high-speed, sub-nanometer-resolution laser interferometer measurements. Such advanced stages allow active monitoring and ultimately active compensation control of stage and instrument vibration (a major contributor to loss of resolution through image blurring). Currently two X, Y and X, Y and rotation capability stages are under development and a new three-dimensional laser interferometer sample stage is in planning.

Ultra-precise laser interferometry is at the heart of Reference Metrology SEMs. Traceability requires the establishment of an unbroken chain of comparisons to stated references. In the case of the Reference SEMs, comparison is made to the meter, the internationally accepted measure of length. Lasers are among the best ways to achieve traceability, but they provide further advantages. Laser systems based on the Renishaw RP 120 type fiber delivery laser interferometry are fast, the built-in flash 12 bit AD converter produces a highest readout rate close to 100 MHz and with 10 MHz the best resolution of 38 pm. They work with a HeNe laser, a homodyne interferometer and a phase sensitive detector. Fiber optics deliver the laser light directly to the measurement axes. Thus beam splitters, mirrors or adjustable mounts are not required and the optical path and complexity, thermal drift and footprint are all reduced. Alignment and installation are simple, the sample stages can be exchanged in a matter of a couple of hours and laser re-alignment can be done in a few minutes. The sample is loaded manually with both Fjeld laser interferometer sample stages. The stages are driven by stepper motors with extremely smooth motion at a stepping distance of 1.24 nm in both the X and Y directions.

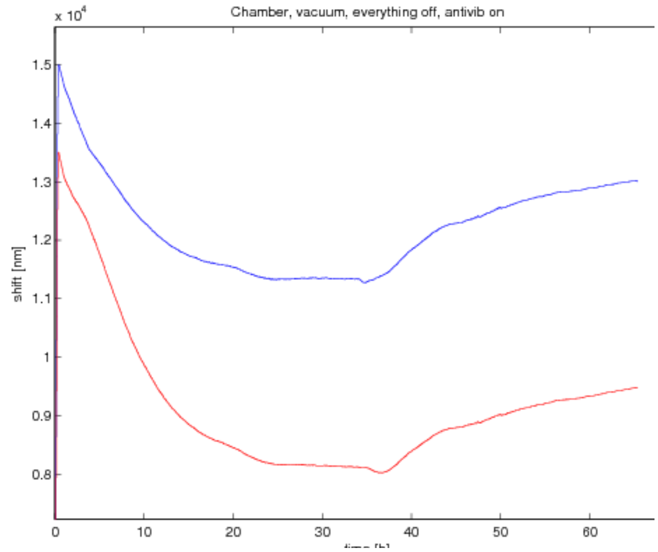

Figure 1. Baseline measurement showing long periods of sub-0.1 nm evenness. Only the anti-vibration platform (concrete slab) and the laser interferometers were at work; 65 hours of data.

The system base-line measurements were carried out to explore the limits of the possible system stability. It was found that the environment and the SEM has a very low noise floor. Figure 1 presents the results of the base-line measurement. For these measurements only the anti-vibration platform (concrete slab) and the laser interferometers were at work. The 65 hour long data show that there are long periods of time, when the system stability has sub-100 pm evenness. Most of the remaining changes come from changes in atmospheric pressure, for which we have sensitive measurement and compensatory methods. All these point to the possibility of excellent stability.

New SEM calibration and High-Performance Standards.

Measurement Methods. These will place the characterization of this most critical of SEM performance metrics on a solid metrological basis and allow objective comparison between instruments and operating conditions, and more accurate digital imaging.

Figure 2 demonstrates the stage motion tracked by the laser interferometer during about 60 seconds of signal collection. Millions of interferometer readings here are organized into a filmstrip to illustrate the typical behavior of the motion of the sample stage. The motion characteristic to all SEM sample stages is essentially a pendulum motion, i.e., the sample stage moves into one direction, slows down, then it

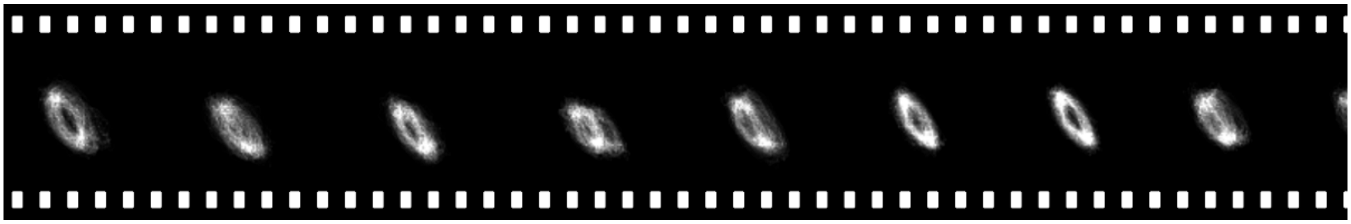

Figure 2. SEM sample stage motion tracked by laser beams for about 60 seconds. Millions of laser readings organized into a filmstrip.

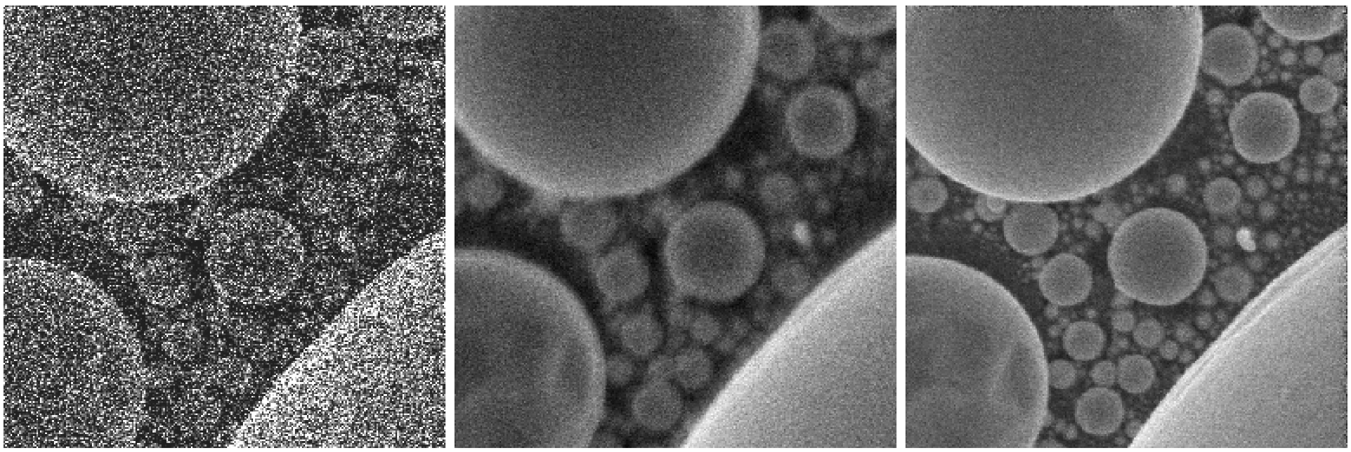

Figure 3. Single image acquired in 11 µs frame time (50 ns pixel dwell time) (left). Traditionally averaged 70 images (middle). The same 70 images averaged with the new, adaptive method (right). The field-of-views are 4.6 µm.

changes its direction, and speeds up again. The motion is not happening in only one direction or plane, and occasionally larger jolts change the overall pattern. The extent of the motion can reach more than 1 nm, which is clearly detrimental to high-resolution imaging. Interferometer-based tracking of the sample stage and compensation for its motions is indispensable for the dimensional measurements of the true nanometer-scale samples and standards.

The consequences of the unwanted, erroneous motions of the sample and the electron beam are severe, because they essentially ruin the resolution and imaging fidelity of nanometer-scale SEM imaging. The best SEMs and HIMs today can readily focus the electron or ion beam into a spot close to 0.3 nm, but in most cases this cannot be fully taken advantage of because the beam never lands at the desired locations because of differential motion between the sample and the beam. Even if the electrons and ions spread within the sample as soon as they hit its surface, it would be advantageous if the focused beam landed within the very close proximity of the intended locations. At the nanometer scale this rarely happens, but the sample motions can be followed by fast laser tracking and the motions of the sample can be compensated for. It is also possible to compensate for the unwanted motions of the beam by the high-speed collection of images or image portions and the use of clever sorting algorithms that assign the individual images to their right position within sub-pixel accuracy.

The quickly collected images are noisy; in fact one of the limits of this new method is that without sufficient sample-related image information and fine details, it is impossible to line up the individual noisy images. Very fast image collection and clever image alignment algorithms can achieve significant improvement in the fidelity and quality of the collected data and images. Figure 3 shows the clear advantage of this new technique. Instead of simply averaging the signal of many fast (and inherently noisy) high-magnification and high-resolution images, the Fourier-transform-based algorithm finds the centers of the images and lines them up with sub-pixel accuracy before averaging. The result is a much higher resolution image that has significantly less distortion caused by the motions of the electron beam. This image shows details that are simply not discernible on the traditionally averaged image. Slow acquisition is a wrong way of image collection for

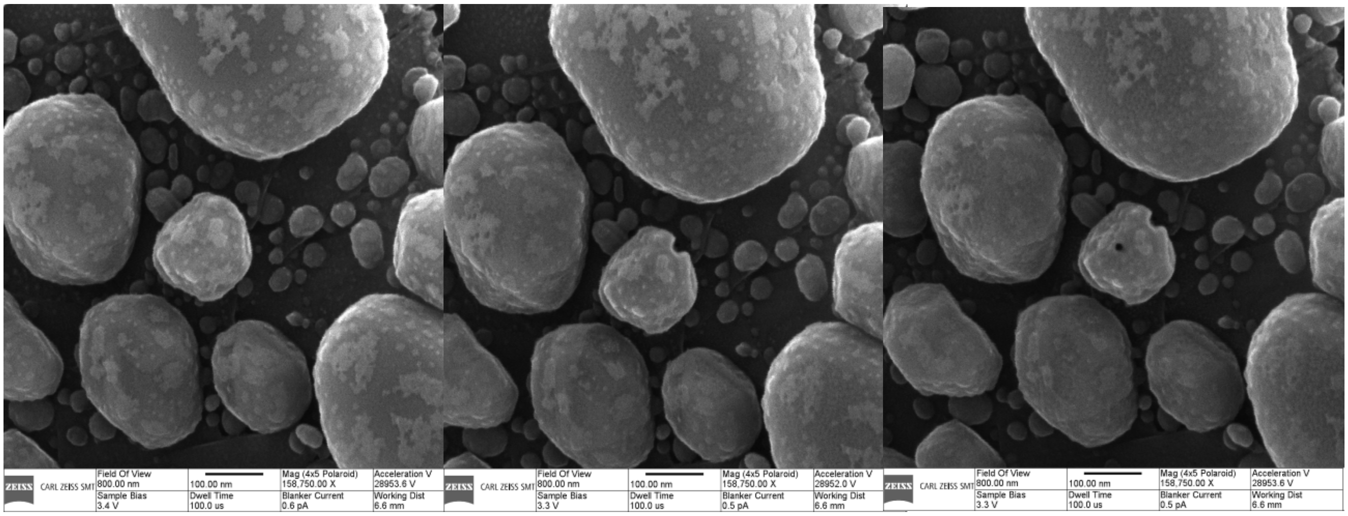

Figure 4. High landing energy HIM SE images of Pt-decorated gold-on-carbon sample (left). After nano-milling (center) and after drilling a 12 nm diameter hole (right). 600 nm field of view images

high-magnification images and data, because it integrates all the unacceptably large disturbances caused by unwanted motions of the sample stage and the primary beam.

Focused Ion Beam (FIB) for High-Precision Ion Milling.

The FIB beyond concurrent imaging allows for the localized, in-situ, simultaneous cross-sectioning and imaging of features of interest for three-dimensional metrology, and for nano-milling with unprecedented resolution.

Figure 4 shows that extremely small patterns can be milled with focused He ion beams at high resolutions. The left image was obtained at the start of the work, and a small square at the two-o'clock position was milled and finally the image on the right was taken after an approximately 12 nm diameter hole was drilled with the He ion beam. This excellent milling capability opens up the possibility to fabricate true nano-structures.

Exploration and Development of Scanning Helium Ion Microscopy (HIM).

HIM metrology and device fabrication is based on new advance in particle beam technique called Scanning Helium Ion Microscope that is analogous to the SEM but uses a probing beam of He+ ions instead of electrons. This results in potentially higher resolution and a greater sensitivity in surface-related information. The work is underway to characterize the sensitivity of the HIM for high-resolution metrology of linewidths in silicon, photoresist, and photomask materials, and to provide to industry (through International SEMATECH) a scientific basis for development of a metrology capability for accurate measurements with the helium ion microscope by modeling ion transport, ion energy loss, and secondary electron production by ions in selected nanomaterials. Another effort has successfully demonstrated the usefulness of the HIM for lithography applications (Figure 5).

Figure 6 proves that very high-resolution imaging in high vacuum, with high landing energy He ions is possible on Cr-on-quartz mask structures. This sample would not yield any acceptable image at the landing energies used for the HIM. The proper adjustment of an electron flood gun makes it possible to compensate for the inevitably appearing positive charging due to ion irradiation.

Establishment of Computational Scanning Electron and Ion Microscopy.

This is an effort to accurately and effectively simulate and model very-high-resolution scanned particle beam images to explore new imaging, image processing and dimensional metrology methods.

Simulation and modeling especially for nanometer-scale SEM and HIM metrology is indispensable. These samples generate much less signal, it is easier to significantly change them by the measurements, so it is imperative to develop measurement optimization methods before the actual measurements take place. Optimized beam and signal collection and processing allow for optimal imaging and the collection of needed information with the minimum extent of sample alteration. Highly sophisticated image

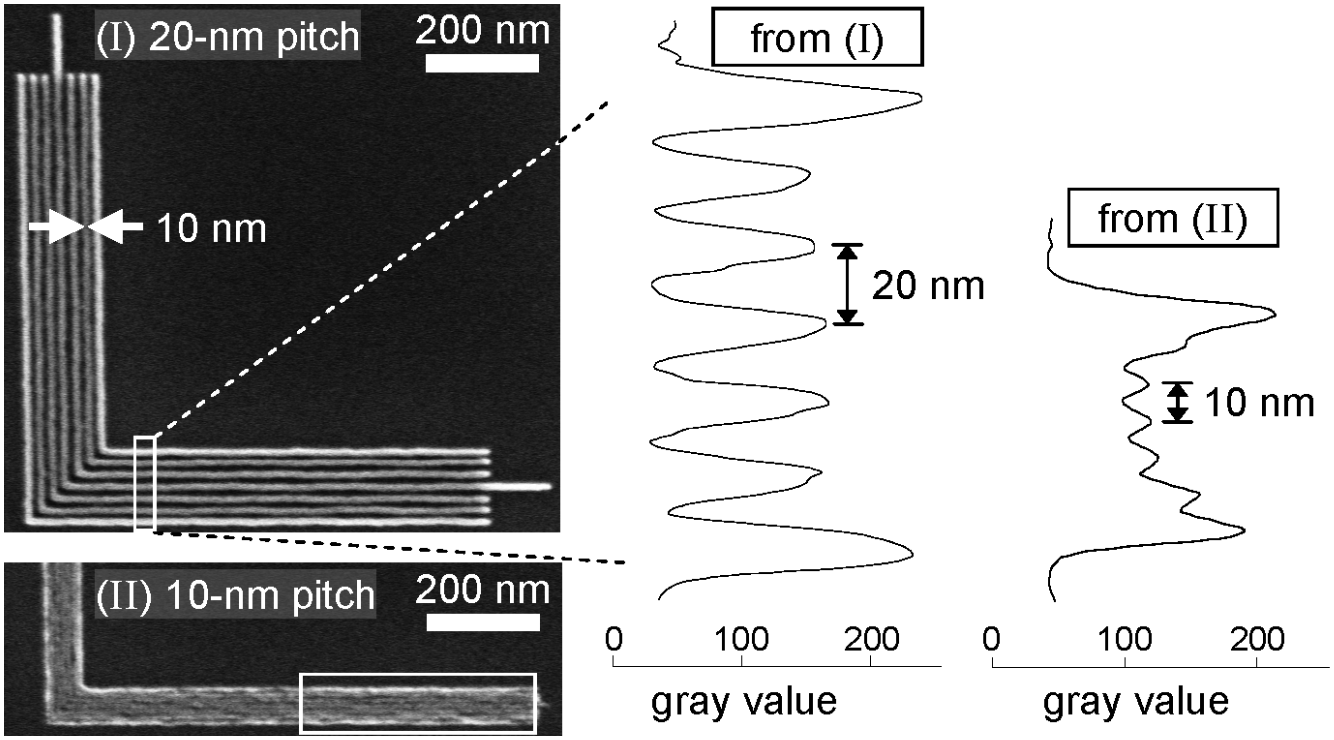

Figure 5. Dense array of 10 nm wide, 20 nm pitch hydrogen silsesquioxane (HSQ) resist lines generated by He ion lithography.

processing techniques that take advantage of the newly available, inexpensive graphical processor-based, essentially supercomputer performance computers is also under development.

Furthermore, new, advanced noise removal methods are also under development. These will remove the noise, but leave the needed, sample-related information undistorted. Figure 7 illustrates the performance of these methods. The assessment of the fidelity of the method is underway.

Fast analogue image simulation is a very useful in the development of new image collection, and processing methods. Thousands of images with precisely known image parameters such as noise, blurring, astigmatism, etc. can be generated in a matter of a few minutes. The method can generate images out of CAD data of integrated circuits or images of essentially any nanometer-size samples. Figure 8 shows a set of images generated for two-dimensional contour metrology method development and optimization.

Impact/Benefit:

- The project is already having significant impact and benefited the semiconductor manufacturing and the emerging nanotechnology industries. The optimized, accurate simulated and modeled measurement methods make it possible to fully take advantage of the high-resolution SEM and HIM instrumentation for scientific and industrial investigations, measurements, and process control.
- NIST expertise is currently being used to improve the performance of the HIM to make it a viable alternative to SEM for nanotechnology and nanomanufacturing.

Accomplishments:

- Developed a strong cooperation with Zeiss regarding the optimization and implementation of He ion scanning microscopy.
- Developed sophisticated new image collection and measurement methods to minimize the effects of vibration in imaging and metrology.
- Developed He ion lithography and nanomilling methods with approximately 10 nm smallest fabricated structures.

Publications:

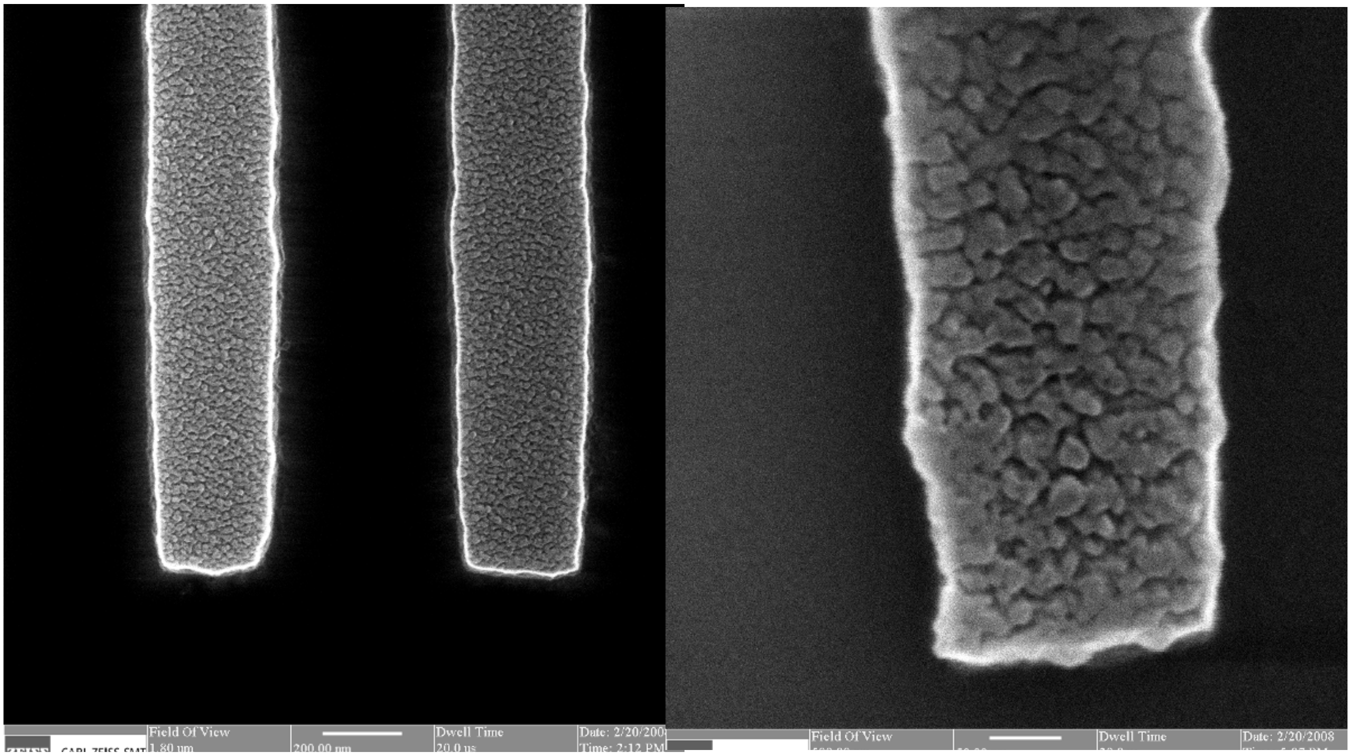

Figure 6. High vacuum, high landing energy HeIM image of Cr-on-quartz mask structures 1.8 µm field of view (left). 500 nm field of view (right).

Book Chapters

- Postek, M. T.,Vladar, A. E., and Ming, B. 2009. Breaking the resolution barrier: understanding the science of helium ion beam microscopy. Frontiers of Semiconductor Research (in press).

Conference Proceedings

- Postek, M. T. Newbury, D., Platek, S.F. and Joy, D. C. 2009. Scanning Microscopy 2009. SPIE Volume 7378, SPIE Bellingham, Washington.

2009

- Winston, D., Cord, B., Ming, B., Bell, D., DiNatale, W., Stern, L., Vladar, A., Postek, M., Mondol, M., Yang, J., and Berggren, C., 2009. Scanning helium ion beam lithography with hydrogen silsesquioxand resist. J. Vac Res (in press).
- Vladar, A. E., Postek, M. T. and Ming, Bin. 2009 On the subnanometer resolution of scanning electron and scanning helium ion microscopes. Microscopy Today 3: 6-13.
- Toth, M., Lobo, C., Laysaght, M., Vladar, A. and Postek, M. T. 2009. Contamination-free imaging by electron induced carbon volatilization in environmental scanning electron microscopy J. Appl. Phys. 106: (unpaged).
- Postek, M. T. Vladar, A. E., Ming, B. 2009. Recent progress in understanding the imaging and metrology using the helium ion microscope SCANNING/SPIE Proceedings 7378 : 737808-1 – 737808-10.
- Cizmar, P., Vladar, A. E., and Postek, M. T. 2009. SEM Image Acquisition with Real-time Blurr and Distortion Correction. SCANNING/SPIE 2009 (in review).
- Postek, M. T., Vladar A. E., and Villarrubia, J. 2009. Nanometrology solutions using an ultra-high-resolution STEM. Hitachi E. M. News May 2009 Number 3 pgs.19-25.
- Cizmar, P., Vladar, A. E., Postek, M. T. 2009. Optimization of Accurate SEM imaging by use of artificial images. 2009 SCANNING/SPIE Proceedings 7378:737815-1 – 737815-6.
- Oral, M.. and A. E. Vladár “Optimization of SEM Imaging through 3D Modeling of Beam and Sample” SCANNING Meeting Monterey May 2009.
- Cizmar,P., A. E. Vladár and M. T. Postek “Image Simulation for Testing of SEM Resolution Measurement” Methods SCANNING Meeting Monterey May 2009

2008

- Postek, M. T. and Vladar, A. E. 2008. Helium Ion Microscopy and its Application to Nanotechnology and Nanometrology. Scanning 30: 457-462.
- Postek, M. T., Vladar, A., Dagata, J.,Farkas, N., Ming, B.,and Wegner, T. 2008. Cellulose Nanocrystals the Next Big Nano-thing? Proc. SPIE 7042: 70420D-1 - 70420D-11.
- Lyons K. W. and Postek, M. T. 2008. Metrology at the nanoscale: What are the Grand Challenges? Proc. SPIE 7042 704202-1 – 704202-13
- Postek, M. T. and Vladar, A. E. 2008. The Potentials of Helium Ion Microscopy for Semiconductor Process Metrology. Proc. SPIE 6922: 69220B-1 - 69220B-7.
- Vladar, A. E., Postek, M. T., Czmar, P. and Villarrubia, J. 2008. Accurate and Traceable Dimensional Metrology with a Reference CD-SEM. 2008. Proc. SPIE 6922: 69220H-1 - 69220H-9.
- Vladar, A. E., Purushotham, K. P., and Postek, M. T. 2008. Contamination Specification for Dimensional Metrology SEMs. Proc. SPIE 6922: 692217-1 - 692217-5.
- Vladar A., Cizmar, P, and Postek, M. T. 2008. Scanning Electron and ion microscope- based size and shape metrology at the nanometer scale. Microsc Microanal 14(2) 118-119.
- Anderson, I. M., Postek, M. T., and Foran, B. J. 2008. EFTEM Spectral Imaging of Fin FET Structure using LACSBI Method ACMM20 and UMAS IV 413-414. Microsc Microanal 14(2) 1186-1187.
- Cizmar, P, Vladár, A. V., Ming, B. and Postek, M. T. 2008. Simulated SEM Images for Resolution Measurement. SCANNING 30:381-391,
- Cizmar, P, Vladár, A. V., Ming,B.,and Postek, M. T. 2008. Artificial SEM images for testing resolution-measurement methods. Microsc Microanal 14(2) 910-911.
- Postek, M. T. et al. 2008. National Nanotechnology Initiative Supplement to the President's Budget 2008. Co-developed by the Nanoscale Science, Engineering and Technology Submmittee, Committee on Technology, National Science and Technology Council (M. Postek participant and contributor) download: http://www.nano.gov.

2007

- Postek, M. T., Vladar, A. E., Kramar, J., Stern, L. A., Notte, J. and McVey S. 2007. The Helium Ion Microscopy: A New tool for Nanomanufacturing. SPIE Nanomanufacturing Conference Vol. 6648, pp 664806-1 – 664806-6.
- Wight, S.,Meier, D., Postek, M., Vladar, A., Small, J., and Newbury, D. 2007. He Ion Induced Secondary Electron and Backscattered Electron Images Compared Side-by-side With Electron Beam Induced Secondary Electron, Backscattered, and Transmission Electron Images. Microsc Microanal 14(2):1186-1187.
- Postek M. T. et al. 2007. The National Nanotechnology Initiative Research and Development Leading to a Revolution in Technology and Industry – Supplement to the President's FY 2007 Budget. Developed by the Nanoscale Science, Engineering and Technology Subcommittee, Committee on Technology, National Science and Technology Council (M. Postek participant and contributor) download: http://www.nano.gov.
- Postek M. T. et al. 2007. The National Nanotechnology Initiative Strategic Plan - December 2007. Developed by the Nanoscale Science, Engineering and Technology Subcommittee, Committee on Technology, National Science and Technology Council (M. Postek participant and contributor) download: http://www.nano.gov.
- Postek, M. T. and K. Lyons. 2007 Instrumentation, Metrology, and Standards Key Elements for the Future of Nanomanufacturing. SPIE Nanomanufacturing Conference 6648, pp 664802-1 - 664802-7.
- Postek, M. T., Vladar, A. E., Kramar, J., Stern, L. A., Notte, J. and McVey S. 2007. Helium Ion Microscopy: a New Technique for Semiconductor Metrology and Nanotechnology. Frontiers of Characterization and Metrology for Nanoelectronics, AIP Press CP931 American Institute of Physics (Seiler et al., eds) 161-167
- Postek, M. T. 2007. The Challenge of Nanometrology. Reprinted and translated into Russian for the Journal of Technical Regulating 2007 #7 pp 8-17 (original publication in Proceedings SPIE Workshop on Nanostructure Science, Metrology and Technology. SPIE Proceedings 4608:84-96.)
- Toth, M.,Lobo, C. J., Knowles, W.R. Phillips, M.R., Postek, M. T., Vladár, A. E. 2007. Nanostructure Fabrication by Ultra-high Resolution Environmental Scanning Electron Microscopy. Nano Letters 7(2):525-530.
- Toth, M., Lobo,C.J., Knowles, W. R., Phillips, M. R., Postek, M. T. and Vladár, A. E. 2007. Nanostructure

Fabrication by Ultra-high Resolution Environmental Scanning Electron Microscopy Microscopy and Microanalysis 2007. 1470-1471CD.

- Cizmar, P., Vladar, A. E. and Postek, M. T. 2007. Image simulation for testing of SEM resolution measurement methods. Scanning 29 (2): 81-82.
- Liddle, A., Vladar, A. E. and Postek, M. T. 2007. Resolution and signal-to-noise measurements in the SEM- a user's perspective. Scanning 29 (2):79-80.

2006

- Londoño, C., Lawall,J., Postek, M. T., Stone, J. and Stoup, J. 2006. The NIST Advanced Measurement Laboratory: At the Leading Edge of Measurement Science and Technology! Proceedings SPIE San Diego 6292. pp 629209-1 – 629209-5.
- Vladár, A. E. Radi, Z., Postek, M. T. and Joy, D. C. 2006. Nano-tip Electron Gun for the Scanning Electron Microscope. Scanning 28:133-141.
- Postek, M. T., Kopanski, J and Wollman, D. 2006. NIST Accomplishments in Nanotechnology. NIST Special Publication 1052, 186pp. and CD.
- Postek, M. T. et al. 2006. National Nanotechnology Initiative Supplement to the President's Budget 2006. Developed by the Nanoscale Science, Engineering and Technology Subcommittee, Committee on Technology, National Science and Technology Council (M. Postek participant and contributor) download: http://www.nano.gov.
- Vladár,A. E., Radi, Z., Postek,M. T. and Joy, D. C. 2006. Nanotip Electron Gun for the Scanning Electron Microscope. SCANNING 28:133–141.
- Postek, M. T. et al. 2006. National Nanotechnology Initiative. NNI Grand Challenge Workshop Report. Instrumentation and Metrology. (Michael Postek, Chairman) (August 2006 available at www.nano.gov).
- Bunday, B.. Banke, B., Archie, C. Solecky, E., Cao G., Hartig, G., Azordegan, A., Postek, M., Villarrubia, J., Vladár, A., Joy, D. and Singh, B. 2006. Unified Advanced Critical Dimension Scanning Electron Microscope (CD–SEM) Specification for sub-90 nm Technology (2005 Version) International SEMATECH Manufacturing Initiative Technology Transfer #04114595B-ENG. 1- 152pp.

2005

- Villarrubia, J. S., Vladar, A. E. and Postek, M. T. 2005. Scanning electron microscope dimensional metrology using a model-based library. Surface and interface Analysis 37:951-958.
- Postek, M. T., Villarrubia, J. and Vladar, A. 2005. Advanced Electron Microscopy Needs for Nanotechnology and Nanomanufacturing. J. Vac. Sci. Technol. B, Vol. 23, No. 6, Nov/Dec 2005 3015-3022.
- Villarrubia, J. S., Vladar, A. E. and Postek, M. T. 2005. A Simulation Study of Repeatability and Bias in the CD-SEM. J. Microlithography Microfabrication and Microsystems. 4(3) 033002-1 - 033002-10.
- Zhang, N. F, Vladar, A.E., Postek, M. T. and Larrabee, R. D. L. 2005. Spectral density-based statistical measures for image sharpness. Metrologia 42:351-359.
- Vladar, A. E., Postek, M. T. and Joy, D. C. 2005. Nano-tip Electron Gun for the Scanning Electron Microscope. MSA Proceedings Microscopy and Microanalysis (supp 2) 766 - 767.
- Vladar, A. E.,and Postek, M. T. 2005. Electron Beam Induced Sample Contamination in the Scanning Electron Microscope. MSA Proceedings . Microscopy and Microanalysis (supp 2) 764 - 765.
- Postek, M. T., Villarrubia, J. and Vladar, A. 2005. Variable Pressure/Environmental SEM a Powerful Tool for Nanotechnology and Nanomanufacturing. Microscopy and Microanalysis (supp 2) 388 – 389.

Customers:

- International SEMATECH and its member companies
- CD-SEM and other SEM instrument manufacturers
- Users of various future standard methods and artifacts across the world

Collaborators:

- International SEMATECH, Advanced Metrology Advisory Group
- International Technology Roadmap for Semiconductors; Microscopy and Metrology Sections
- Zeiss/ALIS Corp.
- FEI Co.
- Hitachi Co.
- E. Fjeld Co.

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Atomic Force Microscope Nanoparticle Metrology

Industry Need:

According to the US Department of Commerce "…the U.S. health care services market is the largest in the world, worth about $1.2 trillion. Rising healthcare costs and ensuring adequate work forces to meet the rising demand for services are key issues…. Costs of health care, labor, prescriptions, medical equipment, insurance premiums and basic supplies continue to rise. Increased insurance premiums are forcing some smaller businesses to drop coverage of their employees." [http://trade.gov/investamerica/health_care.asp] Nanotechnology has been identified by the US National Institutes of Health Cancer Nanotechnology Plan and the US Food & Drug Administration Critical Path Initiative as instrumental for speeding drug discovery, improving efficacy, and reducing costs of pharmaceutical development and manufacturing. This project is working to enable a metrological basis for optimized formulation and regulatory approval of diagnostic and therapeutic nanoparticle formulations.

Project Objective:

The goal of this project is to provide new measurement methodologies that track the formulation, manufacture, and biodistribution of nanoparticles. With nanoparticle production increasing rapidly, there is a critical need for standardized measurement protocols in the field of nanomedicine in which multi-component nanoparticle delivery systems (NDS) target specific cells or tissue, delivering diagnostic and therapeutic agents for the early detection and treatment of disease. Since the structure of these self-assembled entities is often highly sensitive to even slight changes in processing or environmental conditions, dimensional and organizational changes of the constituents can affect the stability and functional performance of the NDS. The Manufacturing Engineering Laboratory at NIST, in conjunction with university and industrial collaborators, has developed a comprehensive suite of scanning probe techniques applicable to the physical metrology of soft, deformable, bio-mimetic materials. This enabling technology is now contributing to the optimization of promising NDS formulations, improving laboratory drug testing, and investigating the health and safety impact of nanoparticles in the environment.

Technical Approach:

We are concerned with measurements of two classes of biomaterials. The first class of measurements seeks to determine the mean size and size distribution of a targeted nanoparticle delivery system (NDS) since they strongly influence the intrinsic stability and functionality of this molecular complex, affects its performance as a systemic drug delivery platform, and ultimately determines its efficacy towards early detection and treatment of cancer. Since its components undergo significant reorganization during multiple stages of self-assembly, it is essential to monitor size and stability of the complex throughout NDS formulation in order to assure its potency and manufacturability prior to entering clinical trials. This work combines scanning probe microscopy (SPM) and dynamic light scattering (DLS) techniques to obtain quantitative and reliable size measurements of NDS and to investigate how variations in NDS formulation or self-assembly process impact the size, structure and functionality of the complex with various therapeutic and diagnostic agent payloads. These combined SPM and DLS methods, when implemented at an early stage of NDS formulation, present a potential measurement approach to facilitate drug discovery and development, optimization and quality control during manufacturing of NDS.

The second class of measurements is performed at the cellular level. Erythrocyte, i.e., red blood cell (RBC), membrane fluctuation mediated by cooperative relationship between its cytoskeleton and lipid bilayer plays an important role in protein dynamics that is indicative of structural-functional properties of healthy or diseased RBCs. Probing of this characteristic membrane behavior requires dynamic interrogation of RBCs under physiological conditions by high-resolution, noninvasive microscopy techniques for which RBCs are required to be immobilized on a substrate while maintaining their viability. Therefore, detailed understanding of the adhesion process and its consequence on RBC shape and dynamic membrane response is critical. In the present study, we demonstrate our ability to engineer substrates with tunable surface zeta potential (SZP) for precise control of RBC adhesion. Specifically, 10 nm gold nanoparticles are adsorbed on poly-L-lysine coated cover slips as a compliant layer to locally modify the non-specific interaction between RBC membrane and substrate. By combining scanning probe microscope (SPM) and differential interference contrast (DIC) imaging techniques we develop a quantitative measurement methodology to investigate the relationship between attachment strength, RBC morphology, cell vibration and membrane fluctuation on these charge and topographically modulated substrates. Adhesion-induced tensing of the RBC membrane on modified substrates leads to changes in cell shape and functionality as determined by SPM force-volume and DIC monitoring of membrane dynamics. The substrate preparation and measurement methods presented here provide a feasible platform to obtain structure-function relationships of viable RBCs under physiological conditions and with that allow us to investigate dynamic behavior of RBCs and their response to diseases.

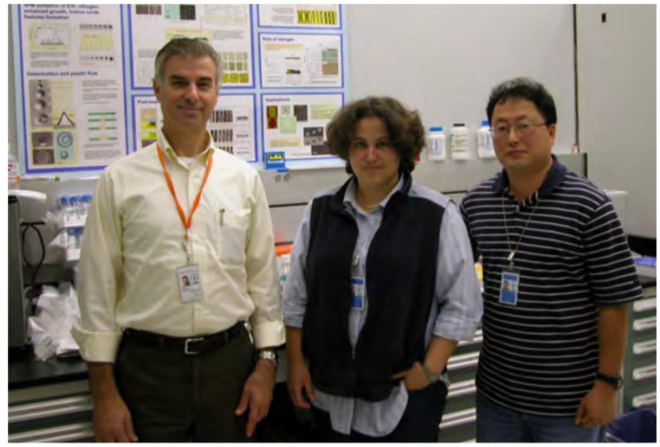

Figure 2. Atomic Force Microscope Nanoparticle Metrology project team members (left-to-right): John A. Dagata, Project Leader; Natalia Farkas, guest researcher; and HyeongGon Kang, guest researcher.

Impact/Benefit:

The Atomic Force Microscope Nanoparticle Metrology Project has developed novel methods for attaching targeted nanoparticle delivery systems to surfaces, leading to more efficient physical optimization of drug formulations. Regulatory approval of NDS formulations by the US Food & Drug Administration is growing. However, standardized measurement protocols for ensuring manufacturability and potency of NDS formulations do not yet exist, thus slowing approval of these promising therapeutic and diagnostic for clinical trials and their availability to patients who could benefit from them.

Currently, drug developers typically use a combination of traditional dynamic light scattering and electron microscopy techniques to monitor and report dimensional aspects of their NDS formulation. However, as used on a routine basis, there is considerable information that is missing about the NDS at the level of individual particles

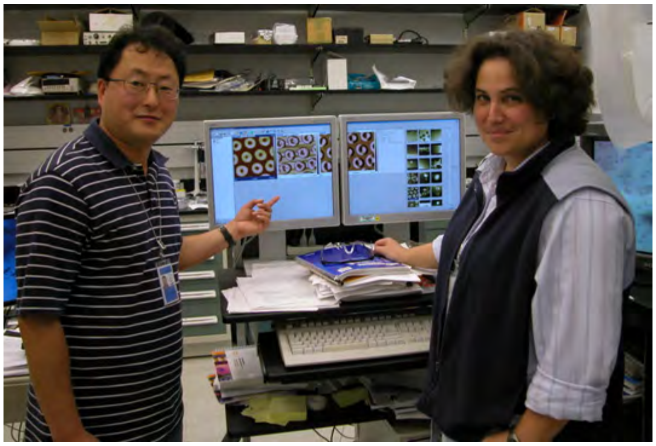

Figure 2. Atomic Force Microscope Nanoparticle Metrology project guest researchers HyeongGon Kang and Natalia Farkas discussing atomic force microscope images of erythrocytes.

as well as from a statistical viewpoint by using only these two techniques. SPM imaging bridges this gap and with further refinement of SPM-based measurement protocols could provide a considerably more effective technique to prepare and characterize nanoparticle contrast agents and therapies for early detection and treatment of disease. The methods developed in this project are being applied to cancer therapeutic and imaging research at the Georgetown University Medical Center, and are contributing to an accelerated path to commercialization for these life-saving formulations.

Accomplishments:

The Atomic Force Microscope Nanoparticle Metrology Project has demonstrated optimized methods for immobilizing intact liposome-based nanoparticle delivery systems (NDS) for scanning probe microscope (SPM) imaging and characterization under fluid conditions. Nonspecific surface binding is an essential first step in high-magnification SPM characterization of nanoparticle delivery systems and their payloads. Since the physical and chemical properties of NDS formulations are dictated by biological and clinical requirements, robust, yet sensitively tunable, methods are required for successful sample preparation. We have identified and implemented a number of new techniques in optimizing the attachment of liposome-based NDS.

This project has evaluated improved magnetic coatings for fluid and dry magnetic force microscopy (MFM) for characterizing dispersed and aggregated magnetic resonance imaging (MRI) contrast enhancement agents. Superparamagnetic iron oxide (SPIO) nanoparticles have become a significant contrast agent for biomedical research. We have applied novel preparation methods to prepare samples for thorough magnetic-based characterization of the particles under dispersed and aggregated conditions. These methods will be applied to evaluate new magnetic materials as well.

The Atomic Force Microscope Nanoparticle Metrology Project has investigated quantitative particle size analysis of gold reference nanoparticles, NDS and SPIOs from SPM images and comparison with dynamic light scattering (DLS). Different approaches to confidently measuring nanoparticles in the 10 to100 nm diameter range depend on considering the material hardness or softness of the sample and taking into account the aspect ratio, height to diameter, of the particles. We have determined particle size distributions by several different methods in addition to SPM imaging in order to understand the factors that lead to variations in the various measurements.

> With nanoparticle production increasing rapidly, there is a critical need for standardized measurement protocols in the field of nanomedicine in which multi-component nanoparticle delivery systems (NDS) target specific cells or tissue, delivering diagnostic and therapeutic agents for the early detection and treatment of disease.

This project has demonstrated optimized attachment of erythrocytes to substrates for combined high-resolution optical and scanning probe microscopy experiments. Protein regulation and cell signaling can be studied by quantum

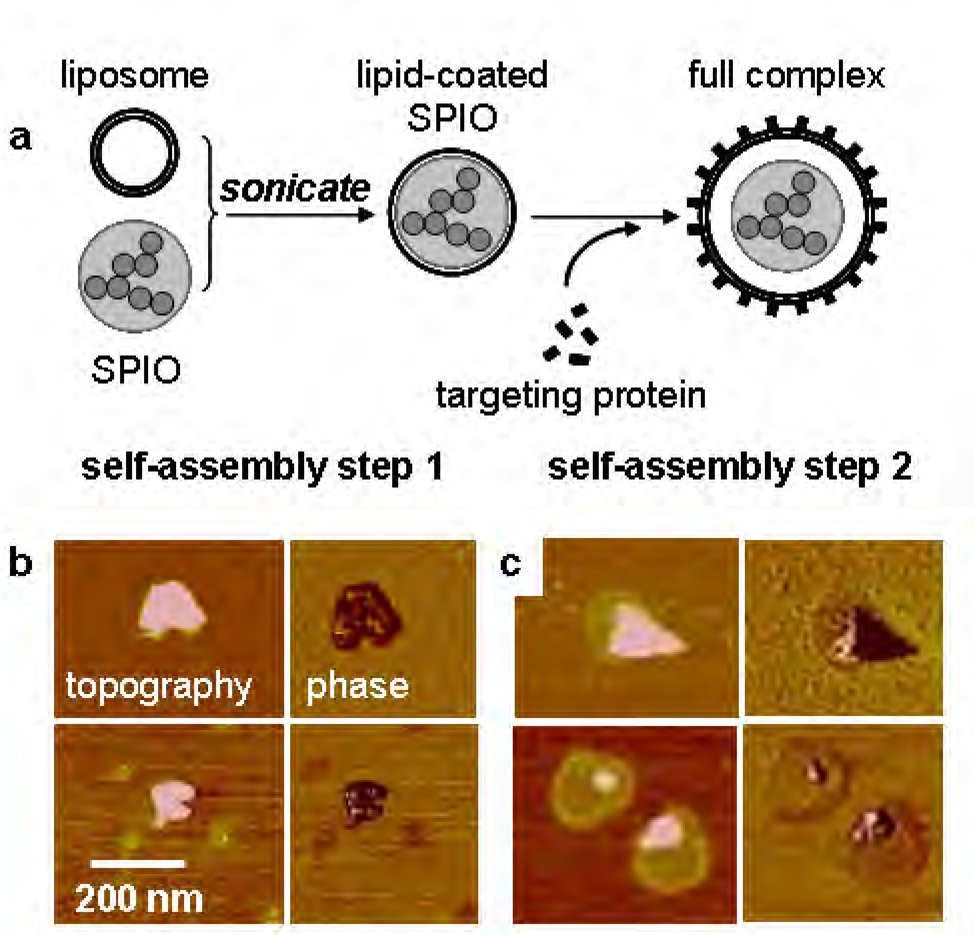

Figure 4. Assembly of a targeted nanoparticle delivery system containing superparamagnetic iron-oxide nanoparticles (SPIOs), a magnetic resonance imaging diagnostic contrast agent. (a) Specific self-assembly scheme optimized for SPIO encapsulation. The process yield lipid-coated SPIOs and full complex after self-assembly step 1 and 2, respectively. High resolution SPM topography and phase images of (b) lipid-coated SPIO and (c) full complex under fluid (upper row) and dry (lower row) conditions. Note that in both fluid and dry images SPIO aggregates are surrounded by a lipid footprint as the full complex is ruptured on freshly cleaved mica.

dot labeling of membrane proteins of living cells; however, cells must be in a viable condition yet sufficiently attached in order to track protein motion along the cell membrane over the course of time. We have validated methodology for doing so and this is being applied to investigate the onset of malaria infection at the cellular level.

Publications:

2009

- Farkas, N., Dagata, J.A., Yang, C., Pirollo, K.F., Chang, E.H. Combined scanning probe and light scattering characterization of multi-stage self-assembly of targeted liposome-based delivery systems, manuscript in preparation.
- Farkas, N., Ramsier, R. D., Dagata, J. A. High-voltage Nanoimprint Lithography of Refractory Metal Films, Journal of Nanoscience and Nanotechnology, in press.

2008

- Yang, C., Rait, A., Pirollo, K. F., Dagata, J. A., Farkas, N., Chang, E. H. 2008. Nanoimmunoliposome delivery of superparamagnetic iron oxide markedly enhances targeting and uptake in human cancer cells in vitro and in vivo, Nanomedicine, 4, p. 318.
- Postek, M. T., Vladar, A., Dagata, J., Farkas, N., Ming, B., Sabo, R., Wegner, T. H., Beecher, J. 2008. Cellulose Nanocrystals the Next Big Nano-thing?, in Instrumentation, Metrology, and Standards for Nanomanufacturing II, edited by Michael T. Postek, John A. Allgair, Proc. of SPIE Vol. 7042, p. 70420D.
- Farkas, N., Zhang, G., Ramsier, R. D., Evans, E. A., Dagata, J. A. 2008. Characterization of zirconium nitride films sputter deposited with an extensive range of

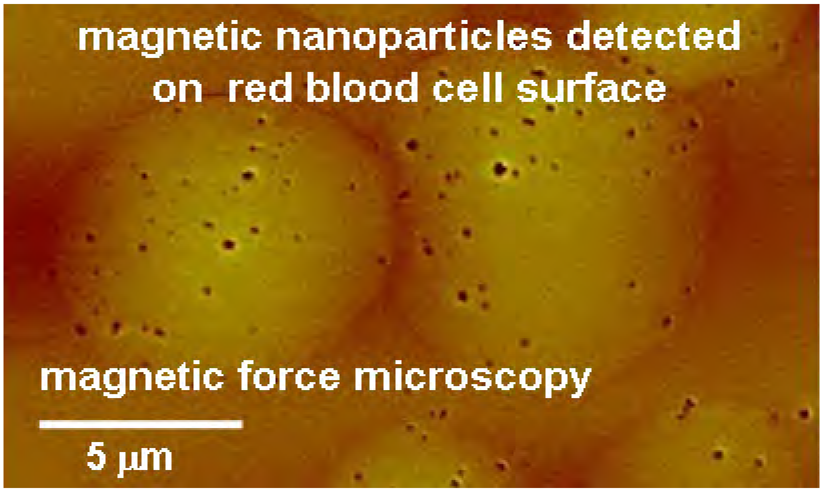

Figure 5. Attachment of superparamagnetic iron-oxide nanoparticle (SPIOs) contrast agent to the surface of erythrocytes. The SPIO aggregates are approximately 50-nm in diameter and consist of elementary particles 10-nm or less. Aggregated magnetic particles are detected sensitively to about 15-nm diameter by magnetic force microscopy in this image.

nitrogen flow rates, JVSTA 26, p. 297.

- Dagata, J.A., Farkas, N., Dennis, C.L., Shull, R.D., Hackley, V.A., Yang, C., Pirollo, K.F., Chang, E.H. 2008. Physical characterization methods for iron-oxide contrast agents encapsulated within a targeted liposome-based delivery system, Nanotechnology 19, p. 305101.

2007

- Pirollo, K. F., Rait, A., Zhou, Q., Hwang, S., Dagata, J. A., Zon, G., Hogrefe, R. I., Palchik, G.,and Chang, E. H. 2007. Materializing the potential of small interfering RNA via a tumor-targeting nanodelivery system, Cancer Research 67, p. 2938.
- Kuramochi H., Tokizaki T., Dagata J.A., Yokoyama H. 2007. Why nano-oxidation with carbon nanotube probes is so stable. I. Linkage between hydrophobicity and stability, Nanotechnology 18, p. 135703.
- Kuramochi H., Tokizaki T., Ando K., Yokoyama H., Dagata J.A. 2007. Why nano-oxidation with carbon nanotube probes is so stable. II. Bending behaviour of CNT probes during nano-oxidation, Nanotechnology 18, p.135704.

2006

- Farkas N., Ehrman J.D., Evans E. A., Ramsier R. D., Dagata J. A. 2006. High-voltage parallel writing on iron nitride thin films, JVSTA 24, p. 1340.
- Rodriguez, O., Fricke, S., Chien, C., Dettin, L., VanMeter, J., Shapiro, E., Dai, H.-N., Casmiro, M., Ileva, I., Dagata, J., Johnson, J., Koretsky, A., and Albanese, C. 2006. Contrast-enhanced in vivo imaging of cancer cells by MRI, Cell Cycle, 5, p. 113.
- Pirollo, K. P., Dagata, J., Wang, P., Freedman, M., Vladar, A., Fricke, S. Ileva, L., Zhou, Q., and Chang, E. S. 2006. A tumor-targeted nanodelivery system to improve early MRI detection of cancer, Molecular Imaging 5, p. 41.
- Dagata, J. A., Fundamental science and lithographic applications of scanning probe microscope oxidation, in S. Kalinin and A. Gruverman, Scanning probe microscopy: Electrical and electromechanical phenomena at the nanoscale, (Springer, NY, 2006) p. 858.

2005

- Farkas, N., Comer, J. R., Zhang, G., Evans, E. A., Ramsier, R. D., and Dagata, J. A. 2005. SPM oxidation and parallel writing on zirconium nitride thin films, J. Vac. Sci. Technol. A 23, p. 846.
- Farkas, N., Comer, J. R., Zhang, G., Evans, E. A., Ramsier, R. D., and Dagata, J. A. 2005. High voltage SPM oxidation of ZrN: Materials for multiscale applications, Nanotechnology 16, p. 262.
- Chien, F.S., Hsieh, W.F., Gwo, Jun, J. S., Silver, R. M., Vladar, A. E., and Dagata, J.A. 2005. Characterization of Prototype Silicon Pitch artifacts by Scanning Probe Oxidation and Anisotropic wet Etching, J Vac. Sci. Technol. B 23, p. 66.

Presentations:

Keynote

- "High-voltage nanoimprint lithography of refractory metal films", Tip-Based Nanofabrication (TBN), TBN2008, John A. Dagata, National Taiwan University of Science and Technology, Taipei, TW, Oct 19-21, 2008.

Invited

- "Fluid scanning probe microscopy: From the nanocell to red blood cells", John A. Dagata, National Institute of Materials Science, Tsukuba, Japan on December 11, 2008.
- "Physical characterization methods for development of nanoparticle delivery systems", 4th Annual Symposium American Academy of Nanomedicine, John A. Dagata, Bolger Center Potomac MD, September 4, 2008.
- "Fluid scanning probe microscopy: From the nanocell to red blood cells", John A. Dagata, Department of Earth Sciences, National Cheng Kung University, Tainan, Taiwan, on October 24, 2008 and the Department of Physics, National Dong Hua University, Hualien, Taiwan, on Oct 27, 2008.
- "Immobilization and Characterization of Nanobioparticles", N. Farkas, J.A. Dagata, K.F. Pirollo and E.H. Chang, American Nuclear Society/ ENS International Winter Meeting, Washington, DC, November 2007.

Contributed

- "Size measurement of targeted nanoparticle delivery systems", N. Farkas, National Institutes of Health (NIH) Nanoweek: The Promise of Nanotechnology for Medicine workshop, NIH, Bethesda MD on April 7, 2009.
- "Size measurement of targeted nanoparticle delivery systems", N. Farkas, 16th Annual NIST Sigma Xi poster day, February 11, 2009.
- "Size measurement of targeted nanoparticle delivery systems", N. Farkas, 55th annual AVS National Symposium, Boston MA on October 22, 2008.
- "Physical Characterization of Liposome-Based Targeted Nanoparticle Delivery Systems with Encapsulated MRI Contrast Agents, " N. Farkas, J.A. Dagata, C.L. Dennis, R.D. Shull, V.A. Hackley, C. Yang, K.F. Pirollo and E.H. Chang, 3rd Annual Nanobiology Think Tank, NCI-Frederick, May 2008.
- "Physical Characterization of Liposome-Based Targeted Nanoparticle Delivery Systems with Encapsulated MRI Contrast Agents", N. Farkas, J.A. Dagata, C.L. Dennis, R.D. Shull, V.A. Hackley, C. Yang, K.F. Pirollo and E.H. Chang, 15th Annual Sigma Xi NIST Postdoctoral Poster Presentation, Gaithersburg, MD, February 2008.
- "Size Measurement of Targeted Nanoparticle Delivery Systems ", N. Farkas, J.A. Dagata, V. A. Hackley, K.F. Pirollo and E.H. Chang, Annual Meeting of the AVS, Boston, MA, October 2007.
- "Tuning the Zeta Potential of Poly-L-Lysine Substrates for the Selective Immobilization of Nanoparticles and Biomaterials", N. Farkas, J.A. Dagata, K.F. Pirollo and E.H. Chang, Annual Meeting of the AVS, Seattle, WA, October 2007.
- "Imaging, Characterization, and Manipulation of Nanobioparticles: Fluid Scanning Probe and Surface Zeta Potential Measurements of Targeted Delivery Systems", N. Farkas, J.A. Dagata, K.F. Pirollo and E.H. Chang, 2nd Annual Nanobiology Think Tank, NCI-Frederick, May 2007.
- "Imaging, Characterization, and Manipulation of Nanobioparticles by SPM in Fluid", N. Farkas and J.A. Dagata, 14th Annual Sigma Xi NIST Postdoctoral Poster Presentation, Gaithersburg, MD, February 2007.
- "SPM Nanolithography of ZrN Thin Films: Nitrogen-Enhanced Growth and Hollow Oxide Feature Formation", N. Farkas, E.A. Evans, R.D. Ramsier and J.A. Dagata, Annual Meeting of the AVS, San Francisco, CA, November 2006.
- "Fabrication of Patterned Iron Thin Film and Microfluidic Phantoms for Quantitative
- Assessments in MRI", N. Farkas, R. Aryal, E.A. Evans, R. D. Ramsier, L.V. Ileva, S.T. Fricke and J.A. Dagata, (50th Annual SAS/ACS/AVS May Conference, Cleveland, OH, May 2006).
- "Nano-Stamped Structures for Bio-Template and MRI Applications ", N. Farkas, R. Aryal, E.A. Evans, R.D. Ramsier, L.V. Ileva, S.T. Fricke and J.A. Dagata, Annual Meeting of the Society for Biomaterials, Pittsburgh, April 2006.
- "Patterned Iron Thin Film and Microfluidic Phantoms for Quantitative Magnetic Resonance Imaging", N. Farkas, R. Aryal, E.A. Evans, R.D. Ramsier, L.V. Ileva, S.T. Fricke and J.A. Dagata, Capital Science 2006, Washington DC, March 2006.
- "High Electric Field Nanoimprint Lithography of Metal

Thin Films", N. Farkas, P. Meduri, E.A. Evans, R.D. Ramsier and J.A. Dagata, Annual Meeting of the AVS, Boston, MA, November 2005.

- "Multiscale Patterning of Metal Thin Films and Polymers", N. Farkas Graduate Student Award presentation, Annual Meeting of the AVS, Boston, MA, November 2005.
- "Patterned Iron Thin Film and Microfluidic MRI Phantoms", N. Farkas, R. Aryal, E.A. Evans, R.D. Ramsier, L.V. Ileva, S.T. Fricke and J.A. Dagata, Conference on Undergraduate and Graduate Student Research, Akron, OH, November 2005.
- "Patterned Iron Thin Film and Microfluidic MRI Phantoms ", N. Farkas, R. Aryal, E.A. Evans, R.D. Ramsier, L.V. Ileva, S.T. Fricke and J.A. Dagata, The Department of Chemistry Advisory Board Meeting, Akron, OH, September 2005.
- "High-Voltage Oxidation of Sputter-Deposited Zirconium Nitride Thin Films", N. Farkas, J.R. Comer, G. Zhang, E.A. Evans, R.D. Ramsier and J.A. Dagata, Microscopy Society of Northeastern Ohio Spring Meeting, Cleveland, OH, April 2005.
- "SPM Oxidation and Parallel Writing on Zirconium Nitride Thin Films", N. Farkas, J.R. Comer, G. Zhang, E.A. Evans, R.D. Ramsier and J.A. Dagata, Joint AVS, ACS, SAS Ohio Spring Meeting, Cleveland, OH, January 2005.
- "Electronic and Ionic Processes in Local Oxidation of Titanium Nitride Thin Films", N. Farkas, J.R. Comer, G. Zhang, E.A. Evans, R.D. Ramsier and J.A. Dagata, Annual Meeting of the AVS, Anaheim, CA, November 2004.
- "Dispersive Kinetics in Atomic Force Microscope Assisted Oxidation of Zirconium Nitride", J.R. Comer, N. Farkas, G. Zhang, R.D. Ramsier, E.A. Evans and J.A. Dagata, MRS Annual Meeting, Boston, MA, November 2004.

Customers:

- US National Cancer Institute (NCI), Bethesda MD
- US Food & Drug Administration, White Oak, MD.

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Collaborators:

- Georgetown University Medical Center (GUMC), Washington DC.
- Aparna Biosciences, Rockville MD.
- Nanotechnology Characterization Laboratory (NCL), Frederick MD.
- University of Akron, Akron OH.
- National Institute of Allergy and Infectious Diseases (NIH), Rockville MD.

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Nanoparticle Manipulation and Metrology

Industry Need:

Nanoparticles are the most mature sector of the rapidly growing nanotechnology industry, but methods to select and fully manipulate nanoparticles are still rudimentary or nonexistent. This limits development of nanofabrication processes at every step from sorting and characterizing nanomaterials to prototyping and testing functional nanodevices assembled from heterogeneous components. Industry requires well-engineered solutions for nanoparticle manipulation, sorting, and sampling for measurement and processing of nanoparticles, rapid prototyping methods to make 2D nanoparticle arrays, and tools to enable prototyping and testing of fully 3D nanodevices.

Project Objective:

The objective of this project is to advance basic nanomanipulation technology to the point that 2D and 3D structures can be rapidly prototyped and tested to enable efficient development of functional nanodevices. We chose optical trapping as the foundation because it is well developed for micrometer-scale particles, does not suffer from surface adhesion, probe interference or other drawbacks of material probes, is non-invasive and can be used within closed test cells and even biological cells, and be reconfigured during use to manipulate one or many particles with complex shapes simply by commands in the control software rather than modifying the instrument. To achieve this, the Nanoparticle Manipulation Metrology Project has developed an optical tweezers system that traps and fully manipulates nanoparticles in three dimensions and up to six degress of freedom.

An optical tweezers instrument works by focusing an optical beam to a small spot (Fig. 1). Small particles are polarized by the field, and drawn into it if their index of refraction is greater than that of the surrounding medium. Optical tweezers can function in air, vacuum, or fluid media, where we use fluids as a convenient means of nanoparticle transport and to control surface chemistry, charge, etc. Note that we use the terms “optical tweezers” and “optical trap” interchangeably here, although strictly speaking we are referring to a single beam gradient trap.

Trapping of nanowires has been demonstrated, as well as the first full 3D manipulation of nanowires in angle as well as position (Fig. 2). This will make possible the accurate, controlled

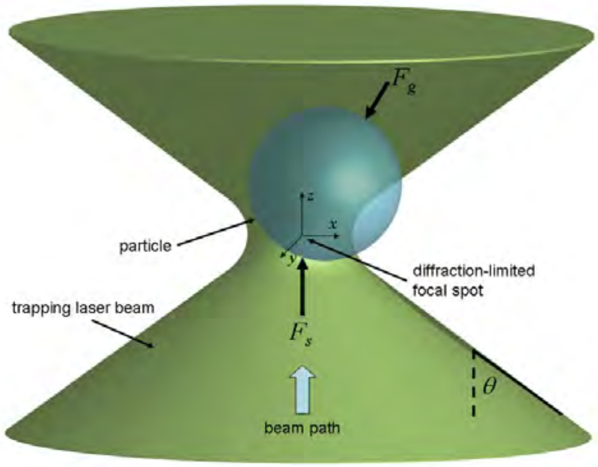

Figure 1. Schematic diagram of optical trap. F_g is the gradient or trapping force, and F_s is the scattering force.

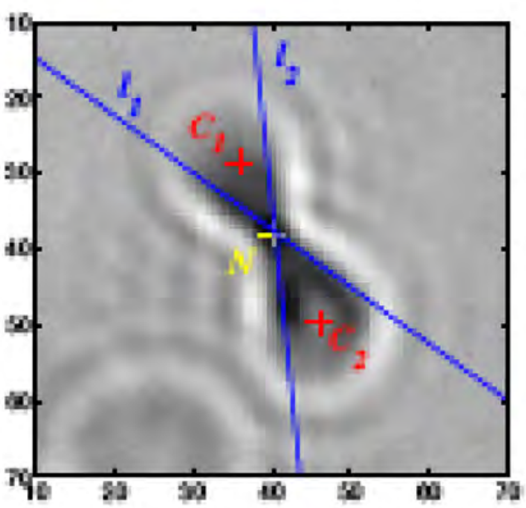

Figure 2. A nanowire trapped along the optical axis, and fixed at an angle.

placement of nanoparticles at predefined locations on substrates, and facilitate the development of accurate particle placement standards for the calibration of optical inspection tools. In addition, the simultaneous trapping of multiple particles in three dimensions has been demonstrated, as well as control of the orientation of non-spherical nanoparticles. This will allow the prototyping and testing of 3D nanostructutes and devices, and the instrument will serve as a testbed for viable assembly methods for nanomanufacturing. Through the controlled placement of nanoparticles, NIST will fabricate an accurate, ordered-array, standard reference material (SRM) for nanoparticle detection and placement.

Technical Approach:

A key aspect of this work is that we leverage the engineering resources of MEL to significantly extend the capabilities of optical trapping, and speed its development as a robust tool. Using custom network-based data acquisition and control software (Fig. 3), the nanoparticle manipulation and metrology project has developed a unique scanning optical tweezers instrument for fast 3D manipulation of nanoparticles with complex shapes, as well as simultaneous manipulation of multiple particles. In addition to 6 DOF manipulation, the system includes a CAD-like interface, support for scripting and automation of measurement and assembly processes, videomicroscopy with custom algorithms for tracking particles in 3D and nanowires in 5D, and fast position measurement with high accuracy.

We have demonstrated a new optical force measurement technique, and the result coupled with the simulation shows that controlled optical trapping should allow nanoparticles to be manipulated much more effectively than is possible today.

The project began by demonstrating the potentially broad applications of optical tweezers by illustrating the wide range of materials that could be optically manipulated including biological nanoparticles used as drug platforms such as liposomes and gold, even though many in the field at the time considered trapping gold to be impossible. Throughout the project a consistent goal has been automation to enable practical nanomanufacturing, which requires accurate location of particles. Therefore vision systems to track micro and nanoparticles were developed in

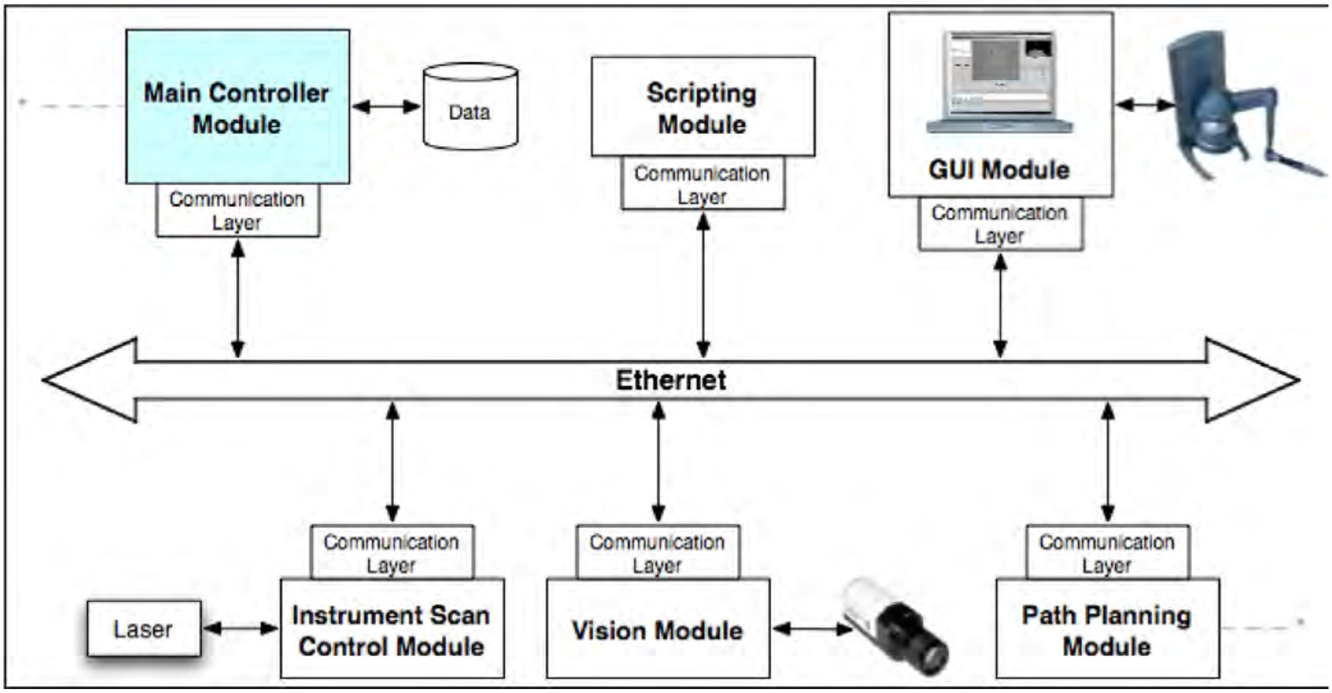

Figure 3. Schematic diagram of the network-based control system that allows the fast trapping instrument to be flexible and expandable.

collaboration with S.K. Gupta's group at the University of Maryland. The systems are able to measure the positions of multiple microspheres in three dimensions from a single image (Fig. 4), as well as distinguish multiple spheres in complex aggregates. The disadvantage of imaging-based systems is the relatively slow bandwidth, so we have also developed high speed single particle tracking to allow the development of fast feedback as described below.

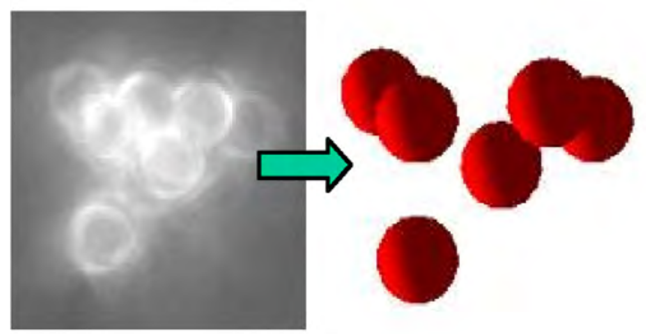

Figure 4. Multiple spheres can also be be distinguished

To augment the set of available nanocomponent building blocks with a more powerful element we extended our system to trap and manipulate nanowires in 5D. Nanowires are important elements because they serve many functions (structural, wave guide, sensor, electronic and photonic) and can be engineered with the desired dimensions and material properties. We fabricated micro and nanowire structures on substrates (Fig. 5), and developed vision systems to completely measure their position and orientation (Fig. 6). Nanowire growth processes are advancing rapidly, but it must be kept in mind that they can be very nonreproducible and that even the wires themselves may not be stable under storage in liquid media even though the bulk material is considered insoluble.

After the first successful manipulation of nanowires in 5D, the project is now developing control systems to push the limits of optical manipulation down to the smallest particles. To prove that control systems can open new capabilities in optical manipulation and nanofabrication, we have developed first-principles simulations to model trapping under laboratory conditions that explicitly include limitations to performance of measurement and control systems. Rigorous simulation of laboratory experiments requires complete measurement of trapping forces because the linear models almost universally used are insufficient and agreement between theoretical calculations and measurements generally poor. We have demonstrated a new optical force measurement technique, and the result coupled with the simulation shows that controlled optical trapping should allow nanoparticles to be manipulated much more effectively than is possible today. Improvements in force measurement, positioning accuracy, and scanning speed are underway that should allow a demonstration of controlled

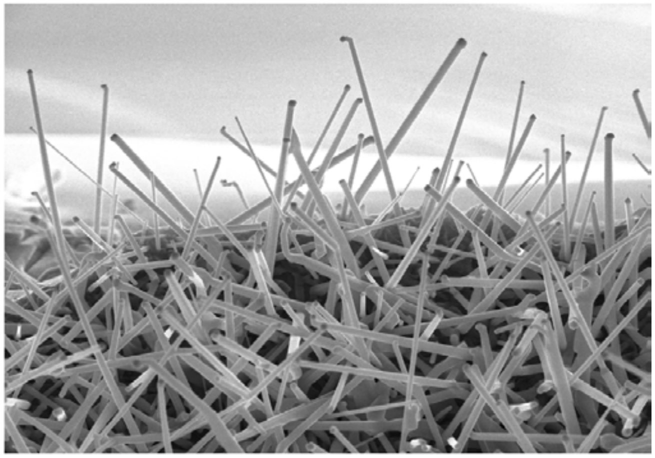

Figure 5. Nanowires as grown and micro and nanowires assembled onto substrates.

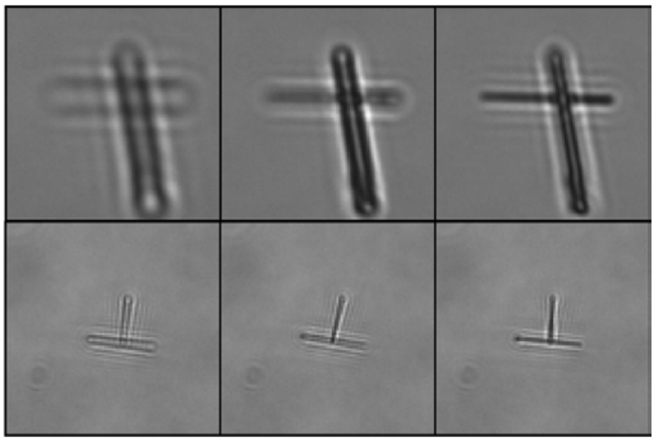

Figure 6. Nanowires manipulated to form a junction.

optical trapping of true nanoparticles in FY10.

Impact/Benefit:

As enabling technology development, the current impacts of this project are in the research sector. The work done in this project has seeded projects at other institutions in the US (UMD, Purdue, UWM) and abroad (Taiwan, China). It has also led to support from NSF and NASA to external efforts, as well as Advanced Technology Program support to multiple laboratories within NIST. The major return on investment will come as the industry need for controlled nanoparticle manipulation finds its application in unique, high-value nanotechnology products.

Accomplishments:

- Developed an integrated scanning trap system for fast 6D manipulation of complex nanoparticles. The network-based system for control and data acquisition allows new instrumental components to extend the functionality and scale to production. In addition to 6 DOF manipulation, the system includes a CAD-like interface, support for scripting of measurement and assembly processes, video microscopy with custom algorithms for tracking particles in 3D and nanowires in 5D, and fast position measurement with high accuracy
- Demonstrated broad applicability of trapping and instrument performance by trapping a wide range of particles including nanowires, and simultaneously trapping red blood cells and pharmaceutical nanoparticles.
- Developed software for video microscopy-based tracking of microparticles in 3D over the full workspace, and nanowires in 5D (x,y,z, + two angles).
- Demonstrated manipulation of nanowires in all five dimensions (x,y,z, angle in plane, angle out of plane). This was the first time this was achieved anywhere to our knowledge.
- Demonstrated suppression of Brownian Motion of microparticles using a control system, as a prelude to controlled optical trapping as discussed above.
- Developed carefully benchmarked first-principles simulations of trajectories of nanoparticles in fluid media subject to trapping forces. This allows controlled trapping to be carefully tested to determine whether the performance of the current generation of control and detection systems is sufficient to allow controlled optical trapping. This determination is crucial because the high rate at which nanoparticles diffuse places great demands on the control system, and accurate simulation is one of the few ways that the practical imitations of control systems (bandwidth, delays, slew rate, resolution) can be accurately predicted. This also allows control algorithms to be compared for optimi¬zation and experimental design.
- Demonstrated new technique to measure optical trapping forces over full spatial range of trap volume. Accurate simulation requires accurate input data, yet optical trapping forces are almost universally measured for micrometer-scale particles using a linear approximation that is only valid near the trap center. As theoretical calculations rarely agree with experiment even within

this simple approximation, we have demonstrated a new force measurement technique that captures the force over the full spatial range of the trap to allow accurate simulation of trapped particle trajectories and instrument performance.

Publications:

2009

- Balijepalli, A., LeBrun, T. W. and Gupta, S. K., "Stochastic Simulations with Graphics Hardware: Characterization of Accuracy and Performance" Journal of Computing and Information Science in Engineering vol. 9(4), 2009
- Gorman, J. J., Balijepalli, A., and LeBrun, T. W., "Control of Optically Trapped Particles for Brownian Motion Suppression." Accepted to be published in the IEEE Transactions on Control Systems Technology, 2009
- Balijepalli, A., LeBrun, T. W., and Gupta, S. K., "Evaluation of a Trapping Potential Measurement Technique for Optical Tweezers using Simulations and Experiments." Proceedings of the ASME Design Engineering Technical Conferences & Computers and Information in Engineering Conference, San Diego, CA, August 2009
- Balijepalli, A., LeBrun, T. W., and Gupta, S. K., "Enhanced Force Measurement Techniques to Extend Optical Trapping towards Nanoscale Manipulation", Proceedings of the IEEE Conference on Nanotechnology, Genoa Italy, July 2009
- Banerjee, A. G., Balijepalli, A., Gupta, S. K. and LeBrun, T. W. "Generating Simplified Trapping Probability Models from Simulation of Optical Tweezers Systems." Journal of Computing and Information Science in Engineering vol. 9(2) pp. 021002-1--021002-9 , 2009

2008

- Balijepalli, A., LeBrun, T. W., Gorman, J. J., Gupta.,S. K., "Methods to directly measure the trapping potential in optical tweezers." Proceedings of the SPIE Optical Trapping and Optical Micromanipulation V, San Diego, CA, August 2008
- Banerjee, A. G., Balijepalli, A., Gupta, S. K. and LeBrun, T. W., "Radial Basis Function Based Simplified Trapping Probability Models for Optical Tweezers." Proceedings of the ASME Design Engineering Technical Conferences & Computers and Information in Engineering Conference, Brooklyn, NY, 2008

2007

- Peng, T., Balijepalli, A., Gupta, S. K., LeBrun, T. "Algorithms for on-line monitoring of micro spheres in an optical tweezers-based assembly cell." Journal of Computing and Information Science in Engineering vol. 7(4) pp. 330-338, 2007
- Peng, T., Balijepalli, A., and Gupta, S. K., "Algorithms forExtraction of Nanowires Attributes From Optical Section MicroscopyImages." Proceedings of the ASME International DesignEngineering Technical Conferences & Computers and Information in Engineering Conference, Las Vegas, Nevada, 2007

2006

- Balijepalli, A., LeBrun, T. W., and Gupta, S. K., "A Flexible System Framework for a Nanoassembly Cell Using Optical Tweezers." Proceedings of the ASME International DesignEngineering Technical Conferences & Computers and Information in Engineering Conference, Philadelphia, 2006
- Peng, T., Balijepalli, A., LeBrun, T. W., and Gupta, S. K.,"Algorithms for on-Line Monitoring of Components in an OpticalTweezers-Based Assembly Cell." Proceedings of the ASME International DesignEngineering Technical Conferences & Computers and Information in Engineering Conference, Philadelphia, 2006

2005

- Lee, D., LeBrun, T. W., Balijepalli, A., Gorman, J. J., Gagnon, C., Hong, D., Chang, E. H., "Development of Multiple Beam Optical Tweezers" Proceedings of the Spring Conference of Korean Society of Precision Engineering (KSPE) pp. 1501-1506, Korea 2005
- Gorman, J. J., LeBrun, T. W., Balijepalli, A., Gagnon, C., Lee, D. "Characterization of optical traps using on-line estimation methods." Proceedings of SPIE vol. 5930 pp. 593023, San Diego, CA, 2005
- Balijepalli, A., LeBrun, T. W., Gagnon, C., Lee, Y., Dagalakis, N. "A modular system architecture for agile assembly of nanocomponents using optical tweezers." Proceedings of SPIE, Optical Information Systems III, vol. 5908 pp. 59080H, San Diego, CA 2005

Customers:

- National Cancer Institute (NCI), Nanotechnology Characterization Laboratory (NCL)

Collaborators:

- University of Maryland
- KJIST, Korea
- University of Western Michigan
- Purdue University

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Molecular Measuring Machine

Industry Need:

The emerging nanotechnology industry is in need of nanometer-accurate two-dimensional position metrology of nanometer-scale objects and features. Much hope has been placed in the current explosion of research efforts in nanotechnology and nanomanufacturing. Incredible devices have been proposed in fields as diverse as medicine, information technology, and military applications. Several novel devices have been demonstrated in prototype form or limited initial production, for example artificial noses, molecular electronics memory and logic elements, photonic devices, meta-materials, and biological probes and healthcare applications including medical diagnostics and therapeutics. For these impressive early ideas and successes to mature into a widespread, stable, and profitable economic sector requires that the infrastructural base of dimensional metrology be extended into the nanoscopic regime. Nanometrology is also needed in commerce or trade for quality assurance and communication of specifications and capabilities. Manufacturing requires process control; process control requires accurate metrology. This remains as true as ever at the nanoscale.

Project Objective:

The objective of the Molecular Measuring Machine (M^3) Project is to provide the ultimate metrology capability for feature placement and two-dimensional position metrology over macroscopic distances. This instrument will then serve as the traceability link for industry metrology instruments via artifacts that will be calibrated on M^3, or through the identification and validation of intrinsic standards and the publication of accepted values and best practices.

Technical Approach:

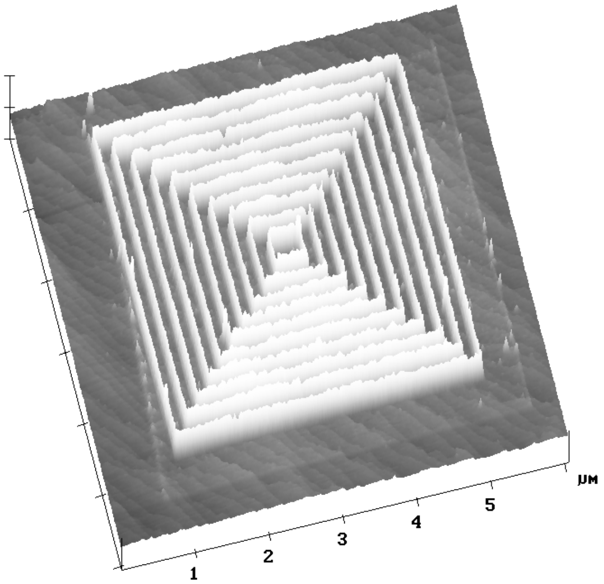
The technical goal of the M^3 project is to achieve atomic-level resolution and measurement accuracy over a macroscopic area. Specifically the goal is atomic resolution feature imaging and one nanometer uncertainty accuracy for two-dimensional feature placement measurements over a 50 mm by 50 mm area. In order to achieve these metrology goals, a scanning probe microscope is used as the sample probe and a Michelson interferometer with a helium-neon laser is used as the metric. The probe is moved with respect to the sample using precision stages, and the measurements are done in a highly-stable, controlled environment.

NIST scientists transferring a sample into the Molecular Measuring Machine vacuum system.

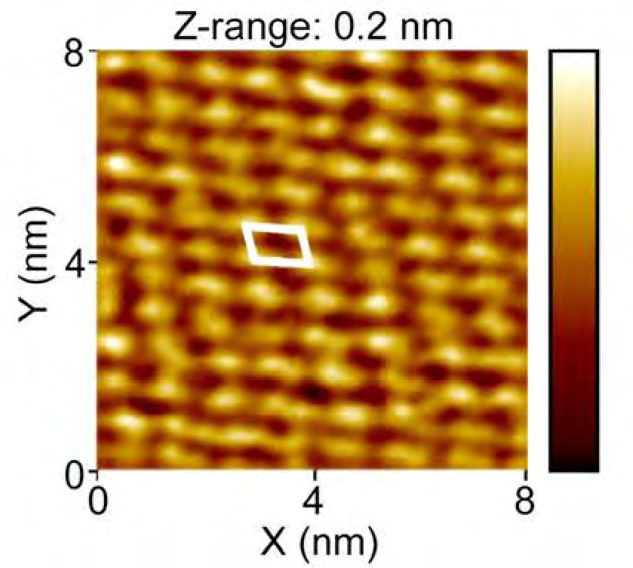
(Courtesy HDR Architecture, Inc./Steve Hall © Hedrich Blessing)

The basic machine core structure of M^3 is a sphere for maximum mechanical stability and stiffness. The X and Y coarse motion is independently generated relative to the core on lower and upper crossed slideways. The X motion carriage holds the metrology reference frame with the specimen inside. The metrology reference frame, or metrology box, has four inside mirror surfaces arranged in a square; the interferometer measurements are referenced to this square. The Y motion carriage carries the scanning tunneling microscope (STM) probe and the interferometer optics. Both the X and the Y motion are generated in two stages, coarse and fine. The coarse motion is driven by stepper motors and guided by the V slideways. The minimum step size is about 500 nm. The fine motion is driven by piezoceramics and guided by vertical, parallel flexures. The fine motion range is 10 µm. The STM probe that is carried by the Y carriage provides the Z axis motion for mapping out the surface topography. It has a Z coarse-motion piezoceramic stepping motor with 300 nm steps and 3 mm range, and a Z fine-motion piezoceramic tube with 8 µm range in the Z axis. No X or Y scan capabilities are provided within the Z actuator, since these motions would not be measured by the metrology system.

The interferometer optics and the STM probe are supported by a common mounting plate on the Y axis carriage. The interferometer system measures the motion of these optics relative to the metrology box that carries the sample. The position of the sample is fixed and unchanging relative to the metrology box during a measurement. The position of the base of the STM probe is fixed with respect to the interferometer optics through the common mounting plate. During a measurement, the STM probe tip moves normal to the mounting plate to track the surface topography. Ideally, this Z axis motion is orthogonal to the X and Y measurement axes defined by the metrology reference frame



Prototype calibration artifact created by M³. Lines are written by scanned probe oxidation on a hydrogen-terminated silicon substrate. The probe is interferometer-guided under closed-loop servo control.



Closed-loop, servo-controlled, nanometer resolution imaging and metrology of a molecular crystal, $((TEET)[Ni(dmit)_2]_2$. a = (1.09 ± 0.07) nm, b = (0.70 ± 0.05) nm, γ = 106° ± 3°.

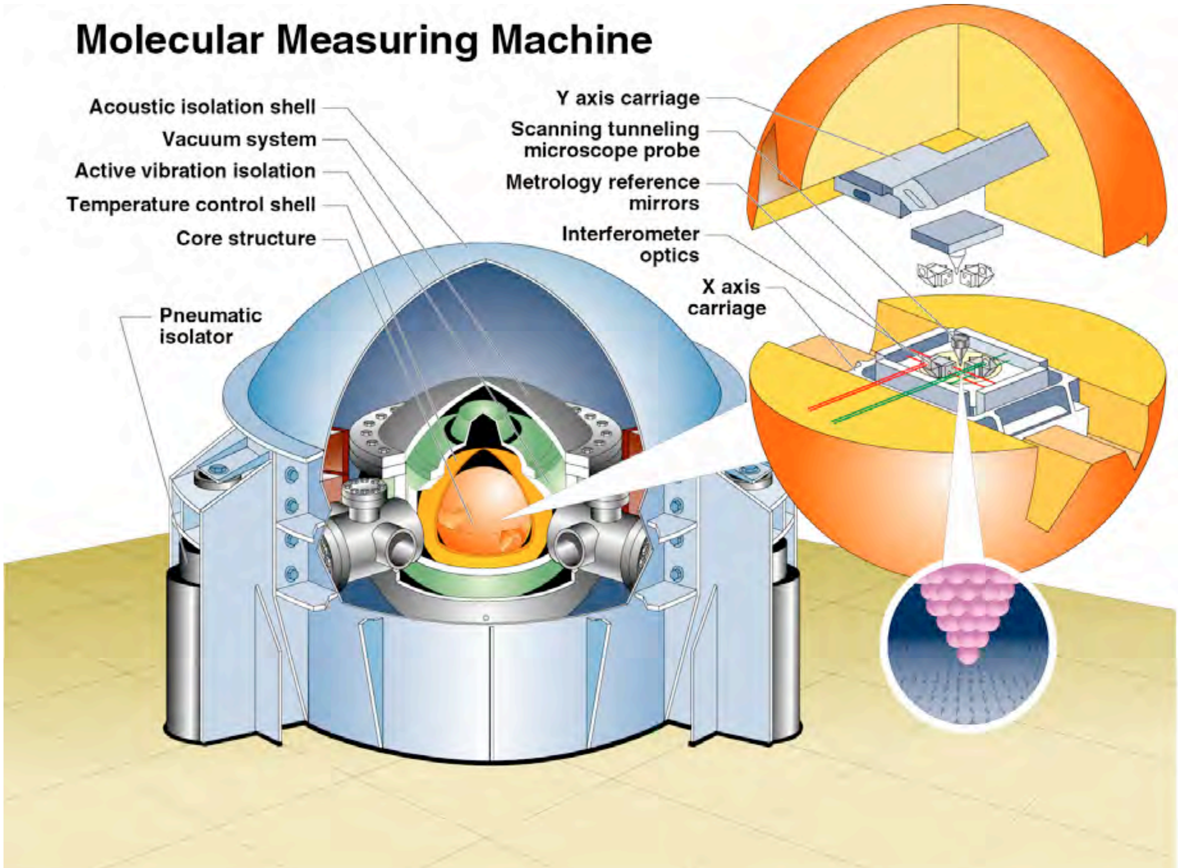
mirrors. The interferometer system uses a differential, two pass, optical configuration resulting in a λ/8 optical fringe spacing, or roughly 80 nm for the helium-neon laser used. Heterodyne fringe interpolation using a time interval analyzer is employed to achieve better than 50 pm least digitization step size at a 5 kHz update rate. The interferometer beam paths are in vacuum to avoid the uncertainties associated with the index of refraction of air.

The M³ machine core is housed within several layers of environmental isolation. There is a radiant heating shroud with the heating current actively controlled to maintain a temperature of 20 ˚C with better than millidegree stability. This assembly is contained in an ultra high vacuum (UHV) chamber capable of achieving 10^{-5} Pa. Surrounding this is a large acoustic isolation chamber with pneumatic vibration isolation legs. There is also a spring suspended vibration isolation stage inside the UHV chamber.

M³ has been applied to the problem of measuring the pitch or line spacing of gratings accurately and precisely. M³ has the capability of imaging sub-micrometer pitch gratings over large distances. This permits the average pitch to be determined to high accuracy. The uniformity of the grating pitch can also be assessed. Metrology instruments that use optical probes typically lack the resolution necessary to distinguish the sub-micrometer pitch features of these gratings. Scanning probe microscopes, on the other hand, typically lack the necessary range and accurate metrology to do a low uncertainty distance measurement. M³ has the capability to do both.

As a specific example, measurements have been made of gratings that were used as reference standards for the grating production process by the Center for Space Research at Massachusetts Institute of Technology for a spectrometer in the Chandra X-ray Observatory of the U.S. National Aeronautics and Space Administration (NASA). In these holographic gratings, the grating pattern is produced by illuminating resist on a substrate by coherent light impinging on the sample from opposite angles thus creating a hologram. The grating pitch is controlled by the frequency of the light and the angle of incidence. The specimens that we measured were roughly 20 mm square. The nominal pitch of the gratings measured were 200 nm for the high-energy transmission grating (HETG) and 400 nm for the low-energy transmission gratings (LETG). M³ was able to measure the average pitch with a relative combined expanded uncertainty (k = 2) of 5 x 10^{-5}.

Measurements have also been performed of the lattice constant of a molecular crystal. In this case, a closed-loop, interferometer controlled scan was achieved with sub-nanometer resolution and position control. The crystal



Cut-away view of the Molecular Measuring Machine instrument.

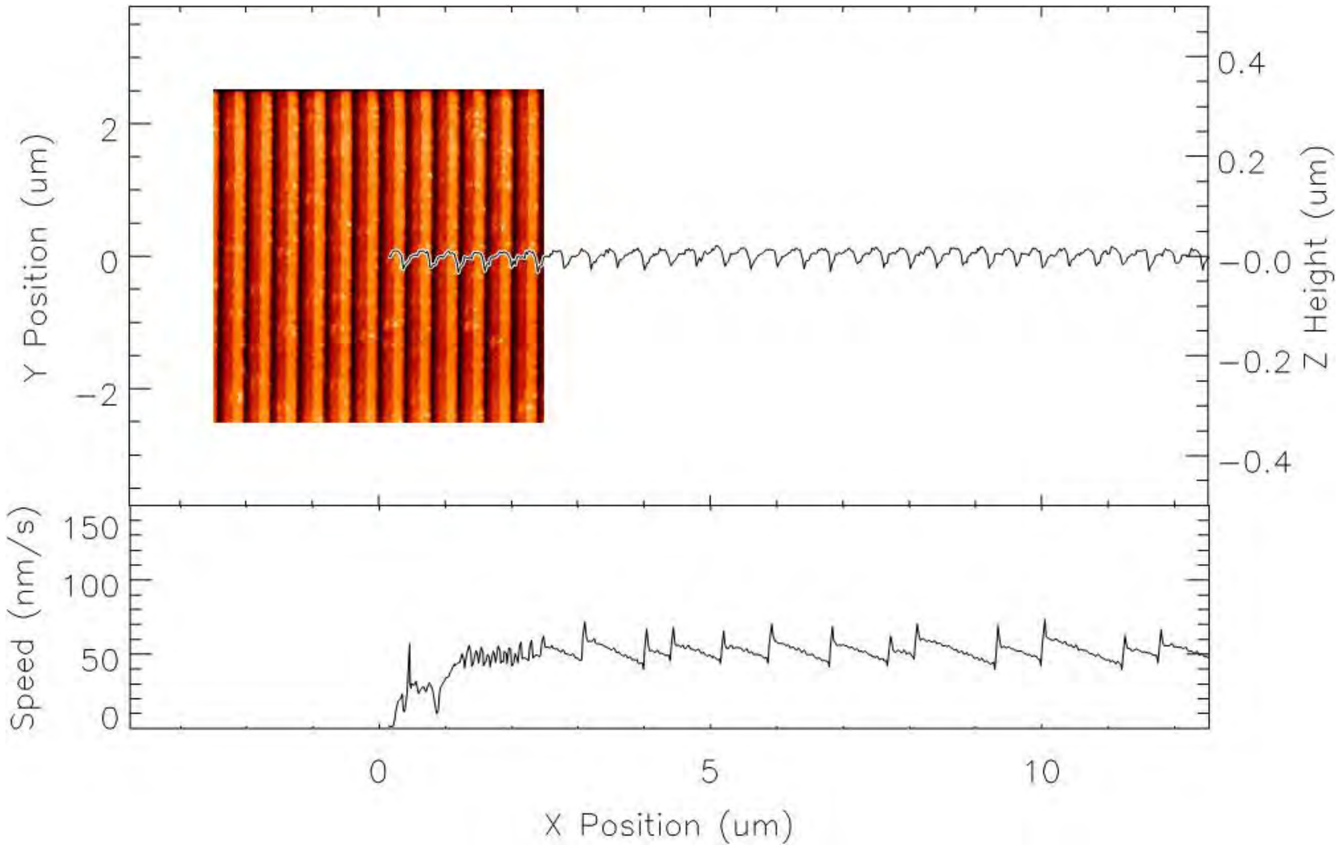
lattice parameters were measured with an uncertainty of only 70 picometers.

Further, the tip-positioning control capabilities of M^3 have been put to use to generate prototype highly precise calibration artifacts. The scanned probe oxidation writing technique, originally developed within the Precision Engineering Division at NIST, was used to create calibration features on a hydrogen-terminated silicon surface. Raising the bias potential on the SPM tip results the local removal of the hydrogen termination and subsequent oxidation of the silicon, which defines a pattern that can be imaged directly, or used as an etch mask to create features with higher aspect ratio. The location of these calibration features can be accurately and precisely controlled via the interferometer system.

Atomic accuracy measurement over a 50 mm by 50 mm area is like dead reckoning on a single dime somewhere in the continental United States.

Impact/Benefit:

- M^3 is providing the measurement traceability path for accurate spectroscopic measurements of NASA's Chandra X-ray Observatory. M^3 measured 200 nm and 400 nm pitch gratings that served as the reference standards for the production of the HETG and LETG (high- and low-energy transmission gratings). All the measurements done on the x-ray spectrometers rely on the M^3 pitch measurements to set the absolute wavelength scale.
- M^3 is laying the groundwork for providing the SI (International System of Units) reference measurements that underpin the use of crystal lattices or other exactly reproducible nanoscale dimensions in nature as intrinsic calibration standards. The measured parameters on the intrinsic standards are a reference value that can be used in the dissemination of the unit of length without shipping a physical artifact, since the user can



For measuring grating pitch with M3, an area can be imaged, measured and analyzed; or a single line profile can be traced for many millimeters distance. Re-imaging the starting location after the line profile corrects for tip wear or drift.

generate their own calibration specimen or obtain one from any source. The length traceability is through an unchanging property of nature, not through an individual measurement of the specific artifact.

Accomplishments:

- Developed a compact, compound actuator and sensor for controlled metrological motion and measurement. This actuator is a novel design that incorporates a 3 mm range coarse motion and an 8 µm range, guided, fine motion actuator with a capacitance gage sensor, all within a $(25\ \text{mm})^3$ volume.
- Developed a method to perform high resolution heterodyne interferometry in vacuum using fiber optic beam delivery. The heterodyne beams are separated for traversal through the fiber optic to avoid mixing and the consequent periodic non-linearity, and then recombined in vacuum. Fiber optic delivery minimizes errors from beam pointing stability and air turbulence.
- Measured the average grating pitch of a laser-focused atomically-deposited chromium grating of 212.69 nm with an estimated expanded uncertainty (k=2) of 5 x 10^{-5}.
- Demonstrated sub-nanometer resolution, closed-loop controlled imaging and metrology based on laser interferometer measurements.
- Demonstrated the fabrication of calibration artifacts using the interferometer system to guide the probe tip, using scanned probe oxidation of hydrogen-terminated silicon as the nanolithography system. The written silicon oxide was subsequently used as an etch mask for reactive ion etching, resulting in a significant enhancement of topographic contrast.

Publications:

- Li, J., Shen, Y.-L., Jeong, J., Scire, F. E., and Kramar, J. A. 2008. A Compact, Compound Actuator for the Molecular Measuring Machine. Proceedings of the ASPE Annual Meeting, 2656

M3 will provide the SI reference measurements that underpin the useof crystal lattices as intrinsic calibration standards.

- Yen, J.-Y., Lan K.-J., and Kramar, J. A. 2005. Active Vibration Isolation of a Large Stroke Scanning Probe Microscope by Using Discrete Sliding Mode Control. Sensors and Actuators A 121, 243–250
- Kramar, J. A. 2005. Nanometer Resolution Metrology with the NIST Molecular Measuring Machine. Measurement Science and Technology 16(11), 2121–2128

Presentations:

- "A Compact, Compound Actuator for the Molecular Measuring Machine," J. Li, Y.-L. Shen, J. Jeong, F.E. Scire, J.A. Kramar, ASPE Annual Meeting 2008
- "Fiber Optic Beam Delivery for Vacuum Heterodyne Interferometery," J.A. Kramar and P. Rachakonda, ASPE Annual Meeting 2005
- "Nanomanufacturing at NIST," J. A. Kramar, American Society of Mechanical Engineers (ASME) International Conference on Manufacturing Science & Engineering, Ypsilanti, MI, Oct. 9-11, 2006. Invited
- "Opportunities in Nanomechanics," J. A. Kramar (Expert Panelist), Frontiers in Mechanical Engineering 2008, at University of Pennsylvania, May 2008

Customers:

- Center for Space Research, Massachusetts Institute of Technology
- National Aeronautics and Space Administration (NASA)

Collaborators:

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Atom-based Dimensional Metrology

Industry Needs:

Nanometrology will play a key role in addressing the challenges in the upcoming nanotechnology industry. A multitude of instrumentation and metrology requirements are foreseeable in the coming years in industrial nanomanufacturing. Industries such as semiconductor manufacturing, flat panel displays, and high density memory requires process control of sub–100 nanometer features at tolerances approaching atomic sizes. Another key challenge in the nano-electronics industry is gate dielectric thickness and roughness requirements, which are reaching the sub-nanometer regime.

"Nanofabrication thus requires the invention of new instruments, measurement tools models methods and standards to characterize nanoscale materials and processes. Only through such developments can the manufacture of commercial volumes of products-with a high degree of repeatability-become economically viable".

--- National Nanotechnology Initiative - Grand Challenge Areas (http://www.nano.gov/html/res/fy04-pdf/fy04%20-%20large%20parts/NNI-FY04_mode2.pdf)

There is a need for specialized metrology tools and processes to meet these requirements. The ultimate limit for nanoscale length metrology is the development of intrinsic calibration standards, where the reference dimensions are based on atom spacing within an ordered, crystalline lattice.

Project Objective:

The continuous miniaturization in manufacturing technologies allows fabrication of nanometer-sized features using advances in precision engineering, lithography techniques and microelectromechanical systems. We must be able to measure and see what we build. Hence atomic scale calibration standards and fabrication techniques have already become an indispensable need for the nanotechnology industry as the critical dimensions of the devices and features gets smaller and smaller. It is also necessary to have atomic scale standards, fabricated and disseminated within the scientific community to enable quantitative calibration and characterization for atomic scale processes. New fabrication atomically precise processes based on nanotechnology will proliferate manufacturing and reach their true potential when the processes become reproducible and reliable. A primary goal of this project is to develop intrinsic calibration standards based on the crystalline lattice.

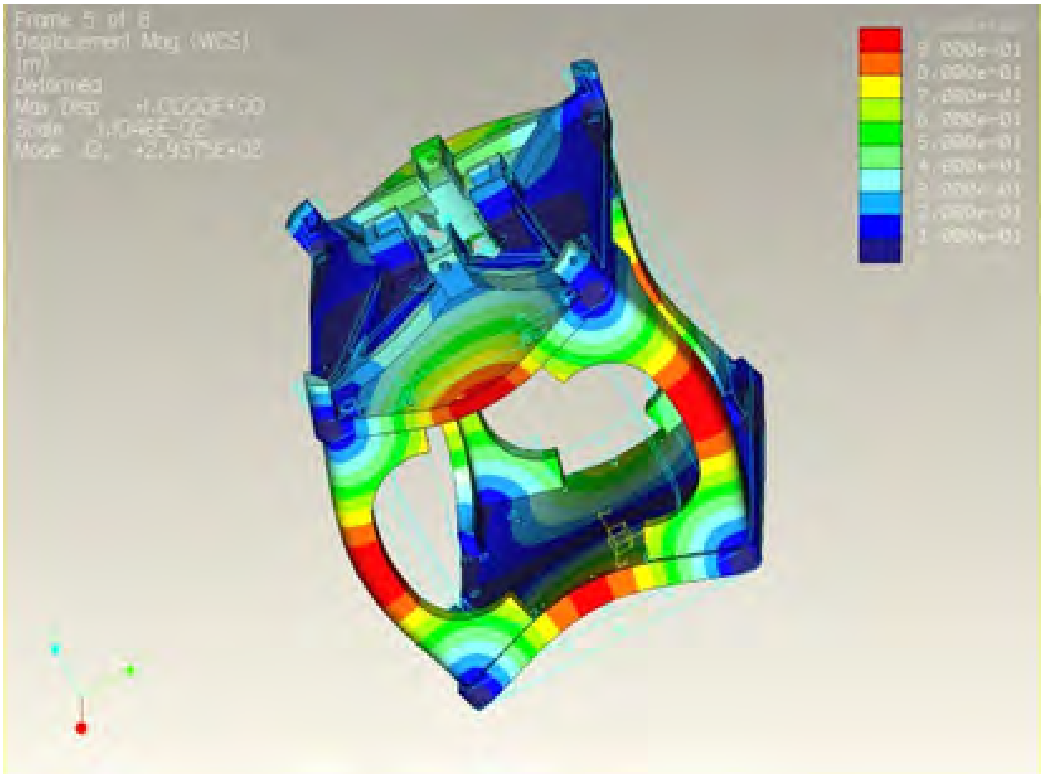

Figure 1. FEA model of the STM Frame to determine the critical modes of vibration

Using techniques developed within this project, advanced step height, linewidth and pitch standards exhibiting picometer accuracy are being developed, using an ultra-high vacuum scanning tunneling microscope (UHV-STM). In addition, a project goal is to validate the use of atomic lattice spacing as a ruler through comparison with interferometric length measurements such as those performed in the Molecular Measuring Machine. The atom-based dimensional metrology project is leveraging this knowledge to develop the underlying measurement science needed to enable parallel, high-throughput, atomically-precise manufacturing for NIST standards development and for a variety of industrial applications.

The project also has a focus on developing methods to prepare photo-lithographically patterned 3D structures in silicon substrates appropriate for atom counting. This includes developing methods for fiducial fabrication to allow overlap between the atomic fabrication domain and the macroscopic world. There are a host of challenges in creating atomic scale features that can be found and accessed using larger scale fabrication and metrology tools.

Another key objective of this project is to achieve parallel high throughput atomically precise manufacturing (APM) using a multitude of parallel STM tips simultaneously. This array of individually activated tips with atomic scale control will then be used to create well-defined atomic features and enable viable throughput for NIST standards development. Hence accurate preparation and characterization of STM probes play an important role in APM where identical and invariant STM probe tips are required for repeatable manufacturing processes and subsequent metrology.

Technical Approach:

Techniques based on scanning probe microscopy (SPM) are used to fabricate three-dimensional surface structures with dimensions ranging from less than 10 nm to 100 nm. These structures are fabricated and imaged using a scanning tunneling microscope (STM). We have designed and fabricated an in-house ultra high vacuum (UHV) STM that implements a Burleigh inchworm/scanner. Since the STM structure is an elastic system it was characterized and optimized by its modes of mechanical excitation and their resonance, see Figure 1. Different modes of the STM frame were detected by mounting the accelerometer at anti-nodes on the structure that were predicted by the FEA results. This analysis enabled us to determine the frequency of the mode that causes bending of the columns which causes fluctuations in the tip-sample gap.

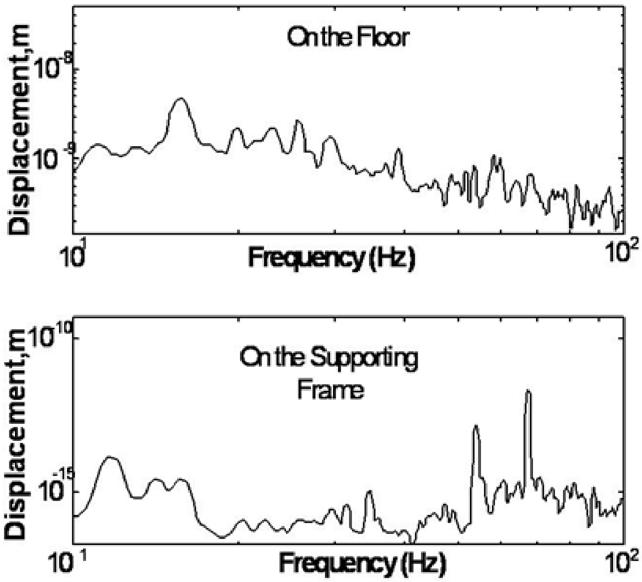

Figure 2: Displacement Magnitude on the Lab Floor and the STM Frame

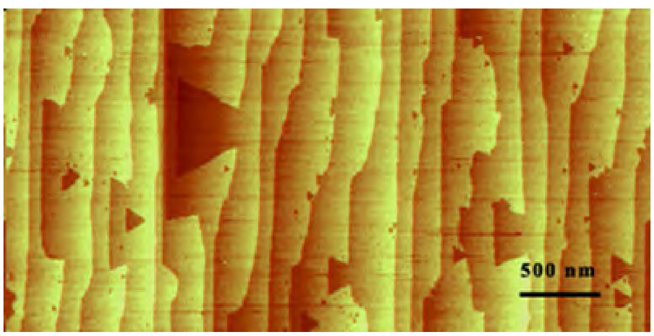

Figure 3. Wet chemically prepared hydrogen terminated Si (111) surface

The STM requires the tip-sample distance to be controlled with picometer precision. This requirement must be satisfied in the presence of external vibrations with micrometer amplitude, temperature drifts and acoustic perturbations. The level of the vibration amplitude at the tunneling assembly should be on the picometer scale, and this is achieved by mounting the entire vacuum chamber on a concrete slab supported on air springs. Figure 2 shows the displacement amplitude spectra measured on the laboratory floor and instrument's supporting frame. We have designed eddy current dampers to attenuate low frequency external vibrations.

We have successfully operated the STM and obtained atomic resolution images. We have recently integrated an RHK SPM controller with the STM. In addition to the in-house designed STM, we employ a commercially variable temperature (VT) UHV-STM (Omicron Nanotechnology) with picometer resolution for imaging as well as performing atomic scale lithography.

In order to fabricate nanometer-compatible photolithographically-patterned three-dimensional structures in semiconductor materials, atomically ordered surfaces must be routinely prepared. We have successfully developed and implemented a wet chemical etching process producing atomically flat surfaces on Si (111) substrates. Figure-3 shows an atomically ordered Si(111) surface with flat terraces separated by single atomic steps. The step height is 314 pm. Exhaustive research was performed to optimize the effects of etching times and wafer miscut on the morphology of etched Si (111) surfaces.

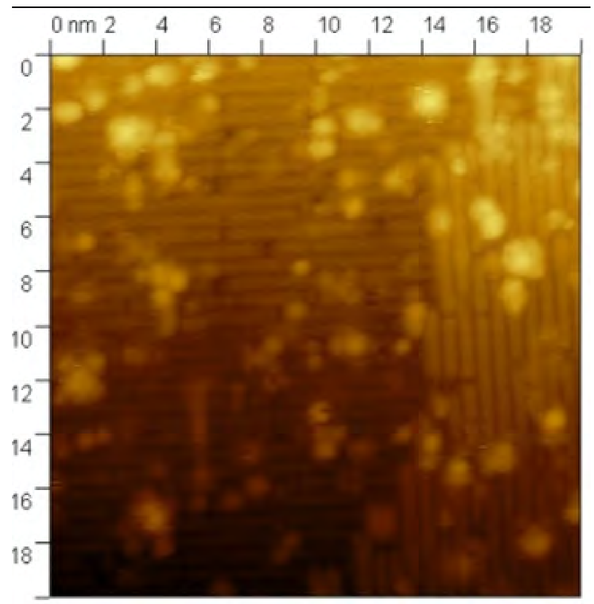

Figure 4. H- passivated atomically ordered Si (100) surface

An alternative approach to routinely produce robust, atomically-ordered surfaces is by thermal processing of silicon. This is being pursued to enable a repeatable, comprehensively controlled, UHV process. An example of a hydrogen terminated repeatable state-of-the-art silicon process using high temperature UHV annealing processes is shown in Figure 4. The project is also focused on developing improved nanofabrication methods using the STM to write atomic-scale features in UHV on hydrogen-terminated silicon substrates using scanning probe oxidation for accurate nano-standard development.

One fundamental goal for the project is to develop methods for repeatably producing atomically-sharp tungsten

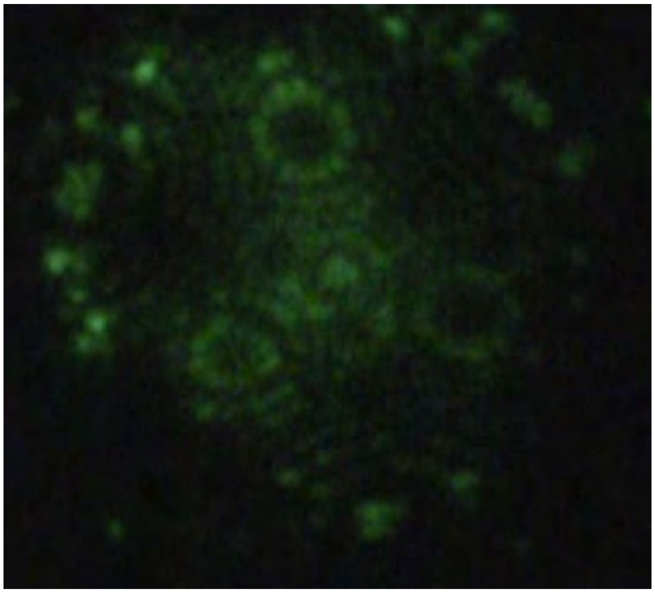

Figure 5: FIM image of a single crystal W(111) tip showing various crystal planes

and alternative-material tips, and evaluate their atomic resolution imaging capabilities. This includes a systematic evaluation of different crystalline materials capable of producing single atom tips or atomic structures defined for high-resolution sub-nanometer imaging. Atomically sharp tips are prepared and characterized routinely from single crystal, polycrystalline tungsten and platinum-iridium wires. Scanning electron microscope (SEM) imaging of electrochemically etched polycrystalline tungsten wire is one routine development tool used. The tips are also characterized with field ion microscopy (FIM)/field emission microscopy (FEM) allowing us to visualize the apex of the tip with atomic resolution, see Figure-5. It is also possible to clean and shape the tip apex by the process of field evaporation wherein the outermost layers of atoms are removed with precise control and accuracy. A routine atomic resolution tip processing procedure is currently being developed in the lab and results using this procedure yield tips that give repeatable atomic resolution STM images.

Impact/Benefits:

- Develop both the metrology for atomic resolution measurements and the ability to fabricate and measure with atomic precision on the nanometer scale
- Develop measurement infrastructure for atomically-precise nanomanufacturing
- Major collaboration and impact on the DARPA funded effort to develop atomically precise nanomanufacturing.
- NIST-developed tip processing and imaging techniques are being adopted by several of the collaborators in the atomically precise manufacturing domain.
- Several elements of silicon sample preparation have impacted the processes being developed by Zyvex Labs for repeatable atomic scale fabrication.
- A number of invited presentations on methods for developing robust atomic fabrication procedures are impacting the funding agencies and research community in this evolving industrial field.

Accomplishments:

- Awarded a 5 year, 3 phase DARPA contract to conduct collaborative research in atomically precise positioning, patterning and metrology.
- Achieved repeatable silicon processing using high temperature UHV annealing processes
- Preparation and in-situ characterization of STM tips to obtain routine atomic resolution STM Images
- Fiducial mark design and reticle fabrication completed
- Studied the effect of etching times and wafer miscut on the morphology of etched Si (111) surfaces
- Implemented Monte Carlo methods to quantitatively study the key aspects of the surface morphology evolution such as step flow, pit expansion, and step-pit collision.
- Demonstrated the first measurement of the surface atom spacing based on a traceable interferometer measurement.
- Designed and built a five chamber ultra high vacuum (UHV) facility regularly capable of producing 1 x 10^{-8} Pa base pressures for performing nanometer scale metrology on various metallic and semiconductor surfaces
- Design of critical components of scanning tunneling microscope (STM) such as such as damping mechanisms for vibration isolation
- Performed modal testing and vibration analysis of the several potential STM frames, STM chamber supports and actual STM mechanical loop structure to improve the mechanical performance of the instrument
- Performed dynamic analyses of the UHV scanning tunneling microscope to study the response of the system to various forms of input vibrations
- Integration of a new RHK SPM controller with the existing Burleigh inchworm based STM.

- Developing a tip processing procedure involving field evaporation and thermal processing to routinely obtain atomic scale resolution images in the STM.

Publications:

2009

- Namboodiri, P., Chikkamaranahalli, S., Li, K., Fu, J., and Silver, R. 2009. Preparation, processing and regeneration of STM tips for consistent atomic resolution imaging. (to be submitted to Applied Physics Letters).
- Namboodiri, P., Li, K., Chikkamaranahalli, S., Atotta, R., Fu, J., and Silver, R. 2009. Self organization and formation of reproducible atomic scale steps and terraces on high temperature annealing of micro patterned silicon surfaces (to be submitted to Applied Physics Letters).
- Li, K., Chikkamaranahalli, S., Namboodiri, P., Fu, J., and Silver, R. 2009. Nanoscale patterning and oxidation on hydrogen terminated Si(100) surfaces. (to be submitted to Nano Letters (ACS Publication).
- Chikkamaranahalli, S., Vallance R., and Silver, R. Optimization of suspension type vibration solators for scanning probe microscopy. (to be submitted to Review of Scientific Instruments).

2007

- Hui Zhou, Joeseph Fu, and Richard M. Silver "A Time-resolved Kinetic Monte-carlo Simulation Study on Si (111) Etching", Journal of Physical Chemistry C, Vol. 111, p.3566, (2007)

2006

- S. Chikkamaranahalli, R. Vallance, B. Damazo, and R. M. Silver, "Damping Mechanisms for Precision Applications in UHV Environment", Proc. ASPE Conf., Pitts. PA, 2006.

2005

- S. Chikkamaranahalli, R. Vallance, B. Damazo, R. M. Silver, and J. Gilsinn, "Dynamic Modeling and Vibration Analysis of a UHV Scanning Tunneling Microscope", Proc. ASPE Conf., Norfolk VA, Oct. 2005.
- Hui Zhou, Joeseph Fu, and Richard M. Silver, "The Influence of Defects on the Morphology of Si (111) Etched in NHF", J Phys. Chem. B, 109, 23386 (2005).

Presentations:

- Kai Li, Sumanth Chikkamaranahalli, Pradeep Namboodiri, Joe Fu, and Richard Silver, "STM-induced Surface Modification with Reactive Ion Etch Pattern Transfer". 53rd International Conference on Electron, Ion, and Photon Beam Technology & Nanofabrication, Marco Island, Florida, May 26 – May 29, 2009

Customers:

- International SEMATECH
- Several University collaborators such as University of Illinois, University of Texas and Austin and Dallas, University of North Texas
- US semiconductor manufacturers
- Zyvex research Labs
- NIST internal nanotechnology researchers

Collaborators:

- DARPA,
- Zyvex Labs Inc.
- SEMATECH
- Intel
- University of Maryland
- University of Illinois at Urbana Champagne
- University of Texas at Austin
- University of Texas at Dallas

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High-Throughput Nanometrology with Scatterfield Microscopy

Industry Need:

Nanomanufacturing requires innovative high-throughput, non-contact metrology methods to minimize defects, quantify critical dimensions, and sample large areas effectively in order for maximum yield. This presents a tremendous challenge, to quantify features and identify defects at the nanometer scale over areas ranging from 1000 µm2 to 1 m2 in dimension. Presently, this requirement is most acutely realized in semiconductor manufacturing. As stated in the latest International Technology Roadmap for Semiconductors (ITRS 2007), one difficult challenge is the development of "nondestructive, production worthy wafer and mask-level microscopy for critical dimension measurement for 3D structures, overlay, defect detection, and analysis." Far-field optical microscopy is unique as it is a high-bandwidth measurement method that is inherently parallel with high-throughput; however its application is impeded by the conventional imaging resolution limit which is proportional to the wavelength of the light. To overcome these limitations, our group has developed scatterfield microscopy, a method that combines the best attributes of high-magnification optical microscopy and scatterometry. While current developments have been implemented to assist the high-throughput needs of the semiconductor industry, high-throughput optical microscopy solutions with nanometer resolution are desperately needed in nascent nanotechnology manufacturing fields such as fuel cell fabrication, nanoparticle metrology, and an array of nanostructured materials.

Looking forward, a growing number of industries will employ nanomanufacturing and nanostructured materials to improve existing products and to create novel devices not yet envisioned. Inherent to all these efforts is the need for accurate, non-destructive nanoscale metrology with high throughput. As manufacturers will increasingly need to maximize accuracy without sacrificing production throughput, some of the best solutions will be found in this new generation of optical microscopy techniques. In some of the most advanced applications of scatterfield microscopy, new measurement strategies using Bayesian statistical methods that nest multiple metrology techniques into the parametric fitting algorithms will provide improved measurement solutions. Meeting advanced industrial measurement needs will require optimizing the tradeoffs among accuracy, uncertainty, throughput, and measurement resolution, which may be best achieved with this new suite of optical measurement techniques.

Project Objective:

The objective of this project is the development of an ensemble of optical microscopy techniques, termed scatterfield microscopy, that enables dimensional metrology beyond traditional optical resolution barriers. The small feature size and resolution barriers make traditional, edge-based imaging metrology impossible. Using new model-based optical methods, quantitative dimensional information can be obtained from the scattered field. By optimizing optical column design, improving experimental methodology, and performing electromagnetic modeling, this project is extending dimensional capabilities beyond previous resolution limits by at least a factor of ten. The goal is to enable the cost-effective mass-production of nanotechnology products through accurate, production capable metrology solutions.

The long-term objective of the project is to yield cost-effective solutions to the metrology requirements intrinsic to the vast assortment of nanomanufactured materials anticipated in the next decade. While many techniques focus on improved accuracy at the nanoscale, this effort uniquely attempts to achieve sub-nanometer resolution over very large fields-of-view. Inherently parallel optical measurements with improved image analysis allow isolation of key information allowing new levels of defect identification and analysis. Ultimately, the challenge is to continuously detect nanometer changes over areas hundreds of square micrometers in size as meters of materials are processed.

This project uses a comprehensive evaluation and investigation for each of the key elements of the optical imaging and illumination hardware to facilitate improved optical imaging and data acquisition techniques. New electromagnetic scattering simulation tools are also used to explore hardware configurations and improve sensitivity to dimensional and materials changes. Measurement modeling, advanced data acquisition algorithms, and optical column design are integrated to achieve new resolution limits.

Technical Approach:

Scatterfield microscopy was developed to meet the next generation manufacturing metrology needs in the semiconductor industry. Image-based optical microscopy techniques have become increasingly challenged in overlay and defect inspection for chip production, and considered not effective for line width metrology. (ITRS 2007) The critical dimensions (e.g. line width) of semiconductor devices decreased below the resolution limit of optical microscopy, forcing the industrial application of other metrology techniques such as scatterometry and scanning electron microscopy. However, resolution limits can be overcome if the information captured in the unresolved image is compared to simulations of the nanostructures being imaged. In its most basic realization, a wavelength and pitch can be chosen that would lead to unresolved lines and trenches when imaged on-axis, but could be resolved by illuminating off-axis, as shown in Fig. 1. In this mode, scatterfield microscopy is a “super-resolution” imaging method. Although in some cases this super-resolution technique may be acceptable, for many more complicated dense structures the technical approach is to replace edge-based metrology (where distances can be determined by applying a metric directly to the image) with signature-based metrology. For the latter, distances are determined from the images by performing parametric fitting against a library of simulation results. When quantitative scatterfield microscopy is performed, the angle of illumination and/or the wavelength of the illumination are varied and the resultant images, or quantities extracted from the images, are compared to those obtained through image simulation,

A critical part of this project is the development and transfer to industry of new optical characterization methods for optical aberration and illumination control using structured illumination.

This departure from traditional optical microscope imaging necessitates the adoption of customized microscopes, or optical columns, tailored to optimize this new high resolution metrology. Two new platforms have been built over the last four years. The first of these is a visible-wavelength microscope operating down to $\lambda = 450$ nm. The optical layout features an open architecture allowing adjustment of virtually all elements in the optical path. The angle of illumination is controlled through the use of Köhler

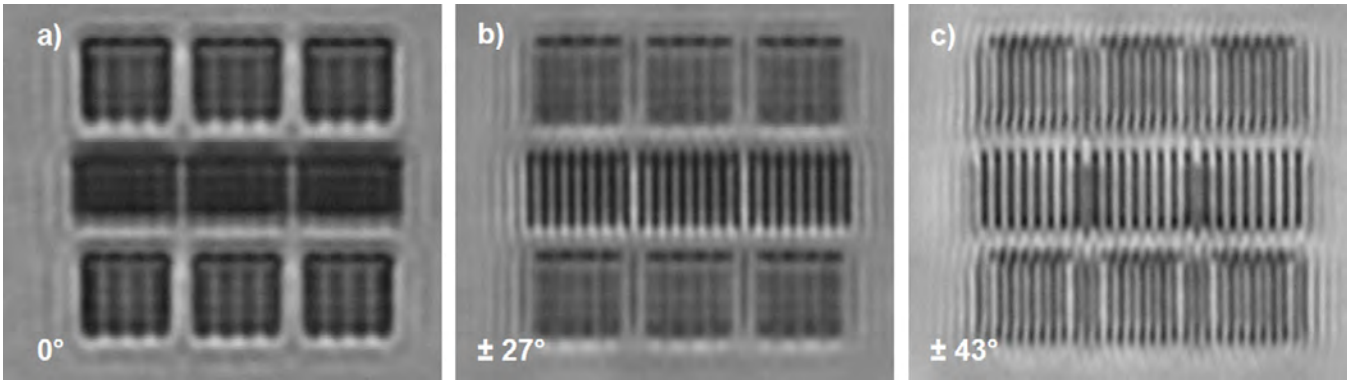

Figure 1. Approximation of images that would be formed by using a physical dipole illuminator, i.e. using two apertures or light sources simultaneously. As our light source is incoherent, we can add two collected images to simulate this effect. (a) Three groups of eight silicon lines appear both at the top and bottom, with three groups of eight trenches imaged in the center row. Features are unresolved with λ= 546 nm. (b) The average of two images collected with the illumination aperture positioned to yield an incident angle of -27° and 27° off the optical axis. Trenches, with pitch p = 434 nm, appear as dark lines. (c) Same as (b), but for -43° and 43°. Both lines (p ≈ 360 nm) and trenches are resolved by rocking a non-zero order of diffraction into the objective lens.

illumination, which provides spatially homogenous intensity at the sample plane. Points conjugate to the back focal plane of the objective in this configuration each illuminate the field-of-view entirely. Therefore, engineering the intensity at this conjugate back focal plane (CBFP) changes the angle of incidence for light illuminating the sample. A pinhole placed in the CBFP dictates the illumination numerical aperture at the sample and moving this aperture off the optical axis changes the incident angle as well. Recent improvements to this tool have enabled a range of visible wavelengths to be used (λ = 450 nm to λ = 750 nm), allowing spectroscopic scatterfield microscopy.

Successful fabrication of the first 450 nm scatterfield tool led to a new advanced second Scatterfield Microscope operating at λ = 193 nm as shown in Fig. 2. The 193 nm tool is actinic and allows the samples to be measured with the same wavelength of light with which they were photolithographically patterned. Reducing the wavelength by more than a factor of two, the resolution of the microscope is substantially improved. This reduction in wavelength is also used to facilitate the measurement of even smaller features using the signature-based Scatterfield approach.

Independent of wavelength, quantitative scatterfield microscopy depends upon the accuracy of the electromagnetic simulations used to analyze these data. The scatterfield optical project has published extensively on improving the correlation between simulation and experimental observations. An in-house model for solving Maxwell's Equations has been implemented based on the Integral Equation Method (IEM). Various models using rigorous coupled wave analysis (RCWA) methods have been comprehensively investigated and validated including both commercially available and an in-house code developed as part of an interlaboratory competence. Additional model comparisons have been completed using three-dimensional finite-difference time-domain (FDTD) and finite-element method (FEM) solutions to the electromagnetic scattering problem. The latter two methods permit the modeling of three-dimensional structures required for some scatterfield microscopy applications. Models are compared to ensure accuracy and also used to develop faster algorithms. [INSERT Fig_3_Best-Fit.TIF HERE] [CAPTION: Parametric fitting for top, middle and bottom line width using angle-resolved scatterfield microscopy. The four colors correspond to combinations of two scan directions (across and along the lines) and two polarization directions (across and along the lines). Simulations are compared to the experimental data in order to determine the best triplet of parameters that yield this optimal fit to the data.]

Process control applications requiring good sensitivity or defect identification implementations of scatterfield microscopy may not require modeling yet may be excellent methods for the detection of particles and defects for nanomanufacturing. Recent applications of this method extend beyond the semiconductor industry into other

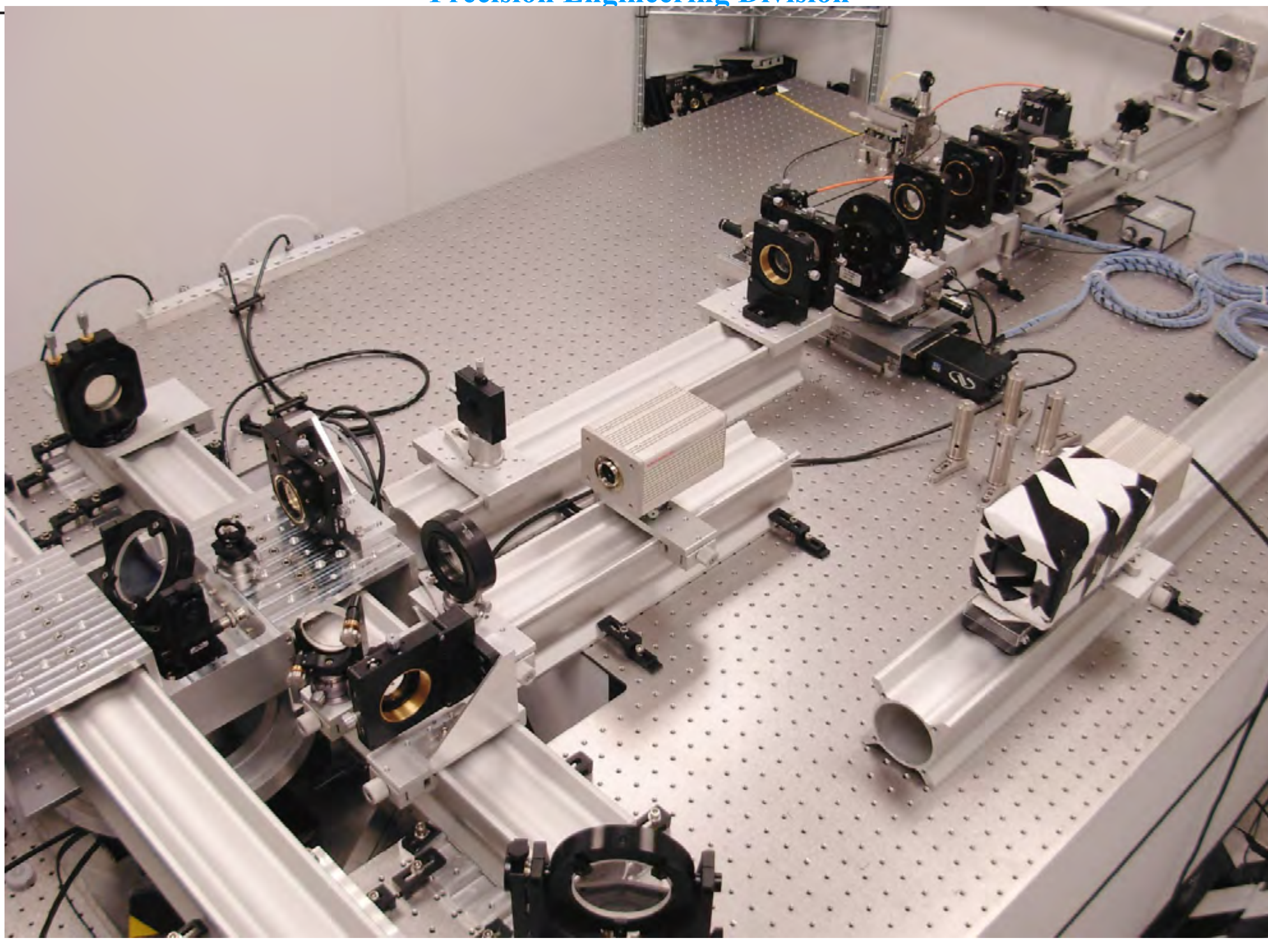

Figure 2. The λ = 193 nm Scatterfield Microscope. Light enters from an excimer laser from outside the clean room, proceeding down the illumination optical column (upper right to lower left corner) to the objective lens (not visible). Light is reflected and sent to one of two collection paths for either high-magnification optical microscopy or Fourier-plane microscopy. In operation, the components are shrouded and purged with N_2 to reduce O_3 production.

fields, including the measurement of particles on fuel cell membranes. Current research topics in this area include establishing intelligent thresholds for defect detection and determining the best wavelengths and angles of illumination for detection of a particular size and type of material.

As the fundamentals of scatterfield microscopy are better understood, a number of improvements have been identified. For example, optimal combinations of angles and/or wavelengths to yield more sensitive measurements can be pre-selected from electromagnetic scattering simulations. Also, advances continue on algorithms for performing these simulations and improving comparisons between experiments and simulation. There has been essential improvement in the accurate modeling of optical measurements leading to never before achieved theory to experimental agreement.

Impact/Benefit:

With significant accomplishments already achieved, the nascent technique of Scatterfield microscopy is already being transferred to industry. The need of engineered illumination and or collection is now recognized by industry (e.g, KLA-Tencor, see M. Adel et al., Proc SPIE 6922, 692202-19 (2008)) and by other national metrology laboratories (e.g. ITRI, see D. Shyu, et al., Proc. SPIE 7272, 72721L 9 (2009)).

Critical dimension metrology requires real time measurement for improved quality control during manufacturing. Similarly, improved high speed defect detection directly affects manufacturing yield. Advances in imaging optics developed within this project are already having a direct effect on these metrology challenges. This project is working closely with semiconductor manufacturers, metrology tool vendors and leading industry

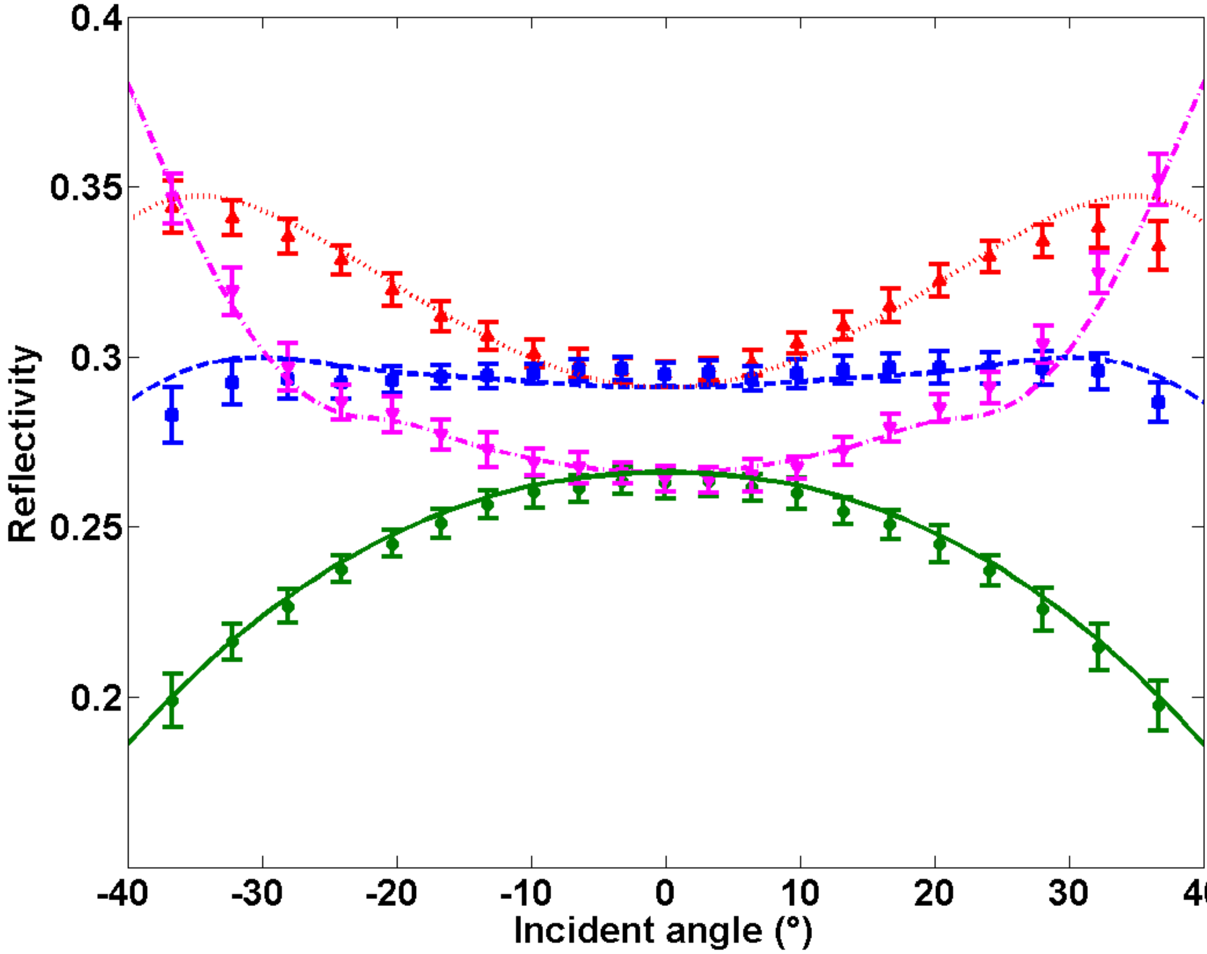

Figure 3. 32 nm defect detection using λ = 193 nm light. At left is the image of a target cell with a "Line end extension" defect. At right is the absolute difference image between this cell and a cell without this defect. This means of comparison is "die-to-database," providing a quick method for isolating probable defects. As these difference signals decrease their optical response will decrease allowing separation of random noise from defect signatures.

consortia to make these new optical methods available as engineering solutions. Another recent example of the transfer of technology developed in this project is the multi-technique nested uncertainty analysis that permits lower uncertainties by combining optics with other metrology tools.

A critical part of this project is the development and transfer to industry of new optical characterization methods for optical aberration and illumination control using structured illumination. This also includes the advanced optical train characterization and normalization methods now recognized as essential for accurate optical microscopy. An important element of this is the test and implementation of a new class of structured illumination using active back focal plane engineered illumination.

A key project output includes completion of the 193 nm Scatterfield optical tool platform and demonstration of full instrument operation in a cleanroom environment with controlled temperature. This includes the extensive application of illumination engineering and optical field control at 193 nm wavelengths. Key elements of tool design, fabrication and alignment have been published and discussed extensively with industrial metrology tool manufacturers and international researchers.

Accomplishments:

- Constructed a dedicated platform for scatterfield (λ = 450 nm)
- Quantitative agreement achieved between rigorous modeling and experimental data. Published results demonstrating quantitative agreement for scatterfield measurements of densely arrayed 100 nm sized lines which vary in 1 nm increments.
- Constructed a λ = 193 nm microscope

- Comprehensive development and comparison of NIST electromagnetic scattering models and industry models completed. Researchers in the optical scatterfield competence have both developed and compared model-based simulation results with excellent agreement.
- Applied the Integral Equation Method (IEM) to characterize profiles
- New approach to evaluation of optical illumination homogeneity, alignment and aberrations developed. This new approach enables evaluation and accurate characterization of optical aberration and illumination homogeneity.
- Implemented rigorous coupled-wave analysis (RCWA) simulations for experiment-to-simulation comparisons
- Correlated parametric fitting of experimental data to reference metrologies
- Extensive parametric modeling was applied. This approach allows evaluation of small changes in complex three-dimensional geometry and optical characteristics. Fractional factorial methods were used extensively to optimize the scatterfield methodology and for improved hardware and instrument design.
- Proposed statistically rigorous method for reducing uncertainties by multi-nested techniques
- NIST competence staff has met with several leading optical metrology tool manufacturers regarding recent advances in scatterfield microscopy. The companies include among others KLA-Tencor, Nanometrics, Soluris, Nova and Applied Materials.
- NIST scatterfield researchers delivered report on optical critical dimension (OCD) limits investigation. NIST researchers are currently funded by SEMATECH to investigate fundamental limits of optical critical dimension measurement techniques.

Publication List:

2009

- R.M. Silver, N.F. Zhang, B.M. Barnes, H. Zhou, A. Heckert, R. Dixson, T.A. Germer, and B. Bunday, "Improving optical measurement accuracy using multi-technique nested uncertainties," Metrology, Inspection, and Process Control for Microlithography XXIII Proc. SPIE 7272, 727202-14(2009).
- B.M. Barnes, H. Zhou, N.A. Heckert, R. Quintanilha, and R.M. Silver, "Spectroscopic Scatterfield Metrology," Frontiers of Characterization and Metrology for Nanoelectronics: 2009 1173, in press (2009).
- R. Quintanilha, Y. Sohn, B.M. Barnes, L. Howard, and R. Silver, "Critical dimension measurements using a 193 nm scatterfield microscope," Modeling Aspects in Optical Metrology II Proc. SPIE 7390, 73900S-12(2009).
- R.M. Silver, B.M. Barnes, H. Zhou, N.F. Zhang, and R. Dixson, "Angle-resolved optical metrology using multi-technique nested uncertainties," Modeling Aspects in Optical Metrology II Proc. SPIE 7390, 73900P-12(2009).
- R. Quintanilha, B.M. Barnes, Y. Sohn, L.P. Howard, R.M. Silver, J.E. Potzick, and M.T. Stocker, "Photomask metrology using a 193 nm scatterfield microscope," Photomask Technology 2009, Proc. SPIE 7488, 74881L-9(2009).

2008

- H.J. Patrick, R. Attota, B.M. Barnes, T.A. Germer, R.G. Dixson, M.T. Stocker, R.M. Silver, and M.R. Bishop, "Optical critical dimension measurement of silicon grating targets using back focal plane scatterfield microscopy," J. Micro/Nanolith. MEMS MOEMS 7, 013012-11(2008).
- R.M. Silver, B.M. Barnes, A. Heckert, R. Attota, R. Dixson, and J. Jun, "Angle resolved optical metrology," Metrology, Inspection, and Process Control for Microlithography XXII Proc. SPIE 6922, 69221M-12(2008).

2007

- B.M. Barnes, R. Attota, L.P. Howard, P. Lipscomb, M.T. Stocker, and R.M. Silver, "Zero-Order and Super-Resolved Imaging of Arrayed Nanoscale Lines using Scatterfield Microscopy," AIP Conf. Proc. 931, 397-401(2007).
- B.M. Barnes, L.P. Howard, J. Jun, P. Lipscomb, and R.M. Silver, "Zero-order imaging of device-sized overlay targets using scatterfield microscopy," Metrology, Inspection, and Process Control for Microlithography XXI Proc. SPIE 6518, 65180F-10(2007).
- R. Silver, T. Germer, R. Attota, B.M. Barnes, B. Bunday, J. Allgair, E. Marx, and J. Jun, "Fundamental limits of optical critical dimension metrology: a simulation study," Metrology, Inspection, and Process Control for Microlithography XXI Proc. SPIE 6518, 65180U-17(2007).

- Y. Sohn and R.M. Silver, "Köhler illumination analysis for high-resolution optical metrology using 193 nm light," Metrology, Inspection, and Process Control for Microlithography XXI Proc. SPIE 6518, 65184V-7(2007).
- R.M. Silver, B.M. Barnes, R. Attota, J. Jun, M. Stocker, E. Marx, and H.J. Patrick, "Scatterfield microscopy for extending the limits of image-based optical metrology," Appl. Opt. 46, 4248-4257(2007).
- Silver, R.M., Attota, R. & Marx, E. "Model-based analysis of the limits of optical metrology with experimental comparisons," Modeling Aspects in Optical Metrology Proc SPIE 6617, 66170W-13(2007).

2006

- B.M. Barnes, L.P. Howard, and R.M. Silver, "Illumination optimization for optical semiconductor metrology," Novel Optical Systems Design and Optimization IX Proc. SPIE 6289, 62890P-11(2006).
- H.J. Patrick, R. Attota, B.M. Barnes, T.A. Germer, M.T. Stocker, R.M. Silver, and M.R. Bishop, "Scatterfield microscopy using back focal plane imaging with an engineered illumination field," Metrology, Inspection, and Process Control for Microlithography XX Proc. SPIE 6152, 61520J-10(2006).
- R.M. Silver, B.M. Barnes, R. Attota, J. Jun, J. Filliben, J. Soto, M. Stocker, P. Lipscomb, E. Marx, H.J. Patrick, R. Dixson, and R. Larrabee, "The limits of image-based optical metrology," Metrology, Inspection, and Process Control for Microlithography XX Proc. SPIE 6152, 61520Z-15(2006).
- Y.J. Sohn, B.M. Barnes, L. Howard, R.M. Silver, R. Attota, and M.T. Stocker, "Köhler illumination in high-resolution optical metrology," Metrology, Inspection, and Process Control for Microlithography XX Proc. SPIE 6152, 61523S-9(2006).

2005

- R.M. Silver, R. Attota, M. Stocker, M. Bishop, L. Howard, T. Germer, E. Marx, M. Davidson, and R. Larrabee, "High-resolution optical metrology," Metrology, Inspection, and Process Control for Microlithography XIX Proc. SPIE 5752, 67-79(2005).

Awards

- "Nanotech Briefs' Nano 50" Awarded to Scatterfield Microscopy Project. The Scatterfield competence was recognized with this respected award with a citation for the development of a revolutionary measurement technique capable of extending conventional optical metrology instrumentation well beyond their current limits.
- SPIE awarded fellowship to Rick Silver in large part for contributions to high resolution optical metrology methods developed under this project.
- Patent Applied for on "Zeroeth Order Imaging" method. This new approach combines standard edge-based imaging with signature-based methods. Only the local zero order scattered light is imaged as a function of illumination angle. Based on simulations, the method is extensible to the sub-20 nm domain.
- Patent Applied for on "Super resolution overlay targets". Joint NIST/SEMATECH patent has been formally applied for on the new super-resolution overlay target, which has the potential to change the target designs and methodology widely used by the industry.
- Several other invention disclosures submitted during this competence. Through-focus focus-metric analysis and imaging have lead to enhanced image technology using information from multiple focus planes. New target designs and optical imaging methods also submitted.

Customers/Collaborators:

- KLA-Tencor,
- Nanometrics,
- Zyvex,
- Applied Materials,
- SEMATECH,
- Intel,
- IBM,
- University of Maryland

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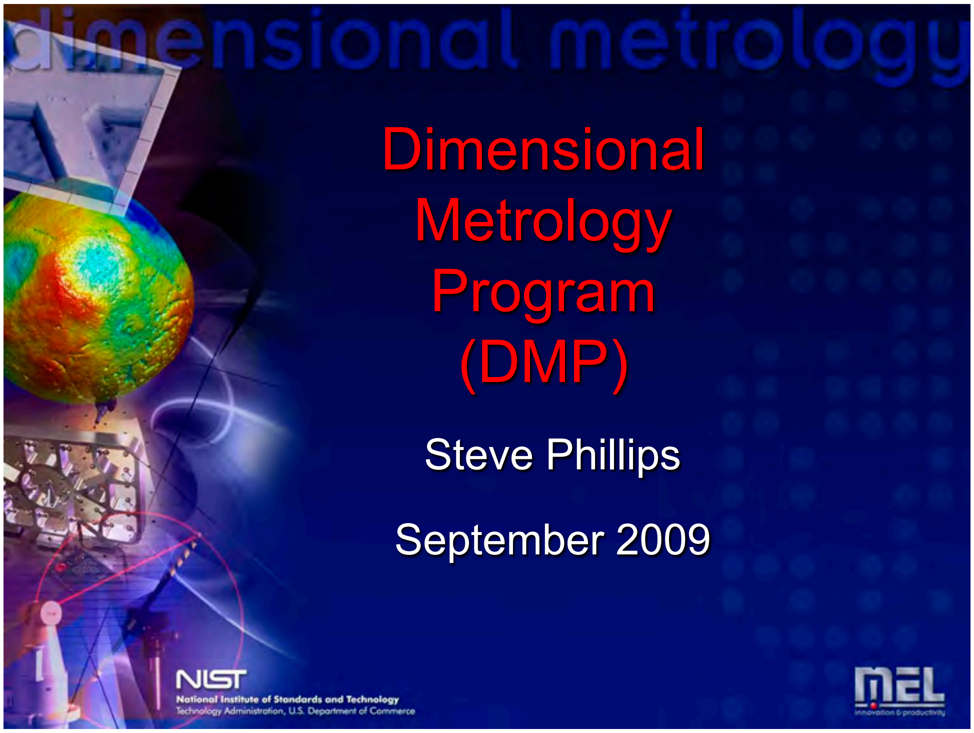
Program and Project Summaries


dimensional metrology
Dimensional Metrology Program (DMP)
Steve Phillips
September 2009
NIST
National Institute of Standards and Technology
Technology Administration, U.S. Department of Commerce
MEL


Overview
About the DMP
DMP Goals (why we do…)
– External: Serving US Industry
– Internal: Developing DMP capability
Long term facilities / capital equipment
Technical Projects (what we do…)


About the Dimensional Metrology Program
The DMP addresses dimensional metrology needs
over nine orders of magnitude: micrometers to kilometers.
The DMP involves :
5 programmatic work projects
– Ted Doiron Quality: PED calibrations + calibration improvements
– Steve Phillips Large scale complex geometry
– Jack Stone MicroFeatures
– Jack Stone Next Generation of the SI Meter
– Rick Silver / Ted Vorburger Surface Metrology


DMP External Goals: Serving US Industry
Improving US industrial competitiveness by:
1. High Leverage
– Providing metrological infrastructure through deep penetration into the US metrology chain
– Developing national & international standards that control specification & testing of entire metrology technologies
Performance Evaluation of Laser-Based Spherical Coordinate Measurement Systems
SURFACE TEXTURE (SURFACE ROUGHNESS, WAVINESS, AND LAY)
Acceptance Test and Reverification Test for Coordinate Measuring Machines (CMMs) — Part 2: CMMs Used for Measuring Linear Dimensions


DMP External Goals: Serving US Industry
Improving US industrial competitiveness by:
2. High Value
– Measurements on high value components
– Measurement on enabling / emerging technologies
Product Life Cycle Curve
Introduction
Growth
Maturity
Decline
SALES
TIME
Ferrule
129 µm
Probe


DMP Internal Goals: Developing DMP capability
1. Developing exceptional capital equipment and facilities to yield unique measurement capability

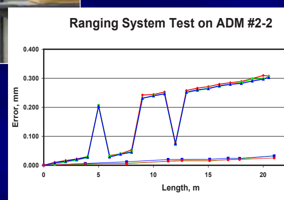
DMP Internal Goals: Developing DMP capability

2. Developing measurement expertise for extremely low uncertainty & difficult to measure measurands

5 year summary of DMP infrastructure

- $9M in new facilities / capital equipment
  - Tape tunnel thermal & measurement upgrade
  - Cooperative & non-cooperative target range
  - Large scale calibration facility
  - Additional M48 CMM
  - New roundness / cylindricity instrument
  - New microprobe for CMM
  - UMAP CMM
  - Optical comb for SI meter realization
  - New surface roughness instrument

Large Scale Complex Geometry Project



Industry Needs Addressed
- Complex surfaces require rapid high density scanning, often over large areas, but no instrument or calibration standards exist.
- The market for this technology is very rapidly growing and US companies are leaders yet no NIST capability exists; measurement traceability is very weak.

Impact/Benefit
- National standards created for coordinate metrology instruments allowing improved industrial capital expenses, lowers barriers to technology adoption, and lowers transaction costs in the marketplace.
- Instrument manufactures are using NIST's calibration facility to improve instrument accuracy and reduce development time and cost

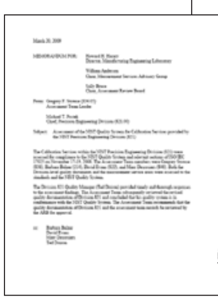
Objective
- Develop calibration facilities, artifacts, and national and international standards for laser trackers, laser scanners, and similar instruments capable of measuring complex geometry workpieces.

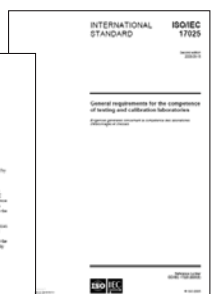
Technical Approach
- Develop calibration facilities for testing a broad class of large scale measurement technology.
- Use detailed mathematical modeling to analyze parametric errors of instruments to optimize calibration and research testing procedures.
- Active participation in national and International standard committees: trackers, scanners, vision technologies.

Highlights / Accomplishments
- World's first laser tracker standard.
- New ISO CMM standard with additional tests and requirements sought by the US .
- Harmonization of US CMM standard with the ISO.
- USCAR flexible coupling project completed sucessfully
- Operational cooperative target calibration facility for ranging measurements to 60 m.
- 800 m^3 large scale volume facility in service

Future Plans
- Autocollimation corrections for range calibration facility.
- Large scale artifact development in progress
- 1D artifact calibration instrument under design

PED Quality System





Industry Needs Addressed
- Calibrations that are not in Appendix C may not be as widely accepted and might not meet traceability requirements.

Impact/Benefit
- Better understanding of customer regulatory environment allow us to better serve our customers needs.
- Traceability for new measurement methods, satisfying ISO 17025 is a requirement.

Objective
•To satisfy international requirements for ISO 17025 based Quality System.

Technical Approach
•Develop PED Quality Management System in conformance with NIST Quality System while preserving the current PED practices and the NIST tradition of personal responsibility as the core of quality management.

Highlights / Accomplishments
•2009 NIST assessment of PED Quality System, findings addressed, and accepted by NIST ARB.
•Partnered with Butler County Community College on technical audit of PED calibrations.

Future Plans
•Performance verification and training new staff will be main focus for the near future.
•Continue partnership with BC3 with technical assessment in spring.

NIST

MEL Precision Engineering Division

PED Calibrations

Industry Needs Addressed
- Increased variety of master gages
- Increased support for calibration of measuring instruments
- Lower uncertainty

Impact/Benefit
- Better control of industrial manufacturing requires lower uncertainty
- Traceability for new measurement methods
- Increased need in support of NIST calibration systems.

Objective
- Increased efficiency of calibrations and support for new industrial measurement methods

Technical Approach
- Deterministic Metrology, using error maps, redundant measurements and statistical process control.
- Upgrade of instruments in standard calibrations
- Increased throughput for non-standard calibrations
- Conformance with NIST Quality System.

Highlights / Accomplishments
- Increased number of high value special tests for NIST projects, industry needs and other government labs.
- Two satellite measurements, small volume, flow nozzles, calculable capacitor.

Future Plans
- Integration of new equipment into standard calibrations.
- Continued training of newer staff on different calibrations.

NIST

MEL Precision Engineering Division

Improvements in Calibrations and Uncertainty Evaluation

Continuous upgrades to the DMP dimensional calibrations maintain state of the art measurement capabilities needed for industrial competitiveness.

Impact/Benefit
- Increased support for new industry measuring instruments – ex. Laser Trackers
- New support for model based calibrations – ex. High accuracy geometry measurements of flow nozzles.
- Standards and explanatory materials to help industry with new requirements for measurement uncertainty budgets.

Objective
- Provide industry with accurate and timely dimensional calibrations to enhance U.S. productivity and quality.
- **Technical Approach**
- Improvement of calibration with new state-of-the-art instruments.
- Better understanding of methods by statistical control and measurement assurance techniques
- Increased outreach to industry through standards activities, conferences and classes.

Highlights / Accomplishments
- Changed paradigm for thermal control from isolation to convective air flow, lowering calibration time and reducing uncertainty.
- New equipment for surface finish, diameter, video based CMM calibrations.

Future Plans
- Second M48 CMM on line to increase throughput.
- New Line Standard Interferometer installed in FY10.
- Revise angle calibrations, replace closure methods with simple comparisons to reduce time and effort.

NIST

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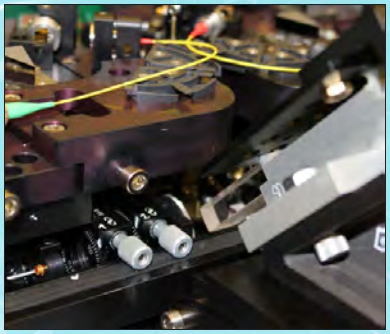
Optical Comb and Refractometry



Industry Needs Addressed
- Multicolor interferometry requires precise knowledge of wavelength beyond simple red lasers.
- Refractive index is the limiting factor for interferometry in air.

Impact/Benefit
- Optical Cavities for refractive index measurement will provide convenient measurements with more than a factor of 2 improvement over current state-of art.
- Comb systems will provide calibration of vacuum wavelength at any frequency, satisfying needs of multicolor interferometry and removing barriers to adoption of novel measurement systems based on new laser sources.

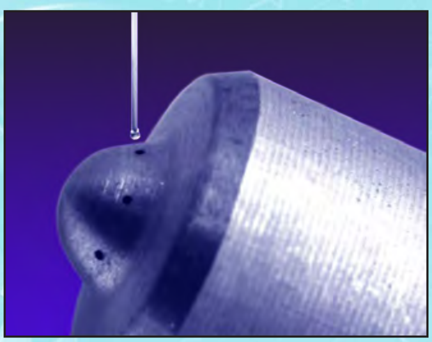
Objective
- Provide verifiable realization of the meter with lasers at any wavelength and with unprecedented accuracy, for improved interferometer-based measurement systems.

Technical Approach
- Optical frequency comb to deliver any desired wavelength at ultra-high accuracy and with clear traceability path.
- Lasers locked to optical cavities for refractive index measurement.

Highlights / Accomplishments
- Flexible tuning of comb and simplified control of offset frequency for robust operation.
- "Length via Satellite": complete suite of procedures for verifiable, traceable operation anywhere in the world using comb + GPS.
- Refractive index: improved optical cavities installed with precise environmental control and lasers locked to track refractive index (Pound Drever Hall).

Future Plans
- Integrate refractometer into high-accuracy dimensional measurement systems.
- Multi-color techniques and absolute interferometry with comb.

Microfeature Calibration Development



Industry Needs Addressed
- Micro-parts play an expanding role in our economy and require precision measurements beyond current capabilities. Areas of potential impact include MEMS, advanced fuel injectors, micro-optics (including optical fiber positioning for telecom), and microfluidics.

Impact/Benefit
- High-accuracy measurements provide data to understand how form/dimension of new devices under development affect function.
- Traceable measurements will support commerce and trade in microfeature parts.

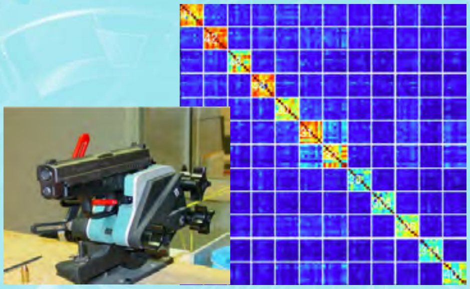
Objective
- Provide new measurement capabilities to industry in the micro- regime: develop and fully characterize instruments and techniques for measurement of microfeatures.

Technical Approach
- CMM-based measurements (M48 or UMAP)
- Micro-probes of several designs: NIST probe, InSituTec, Mitutoyo, Gannon

Highlights / Accomplishments
- Practical measurement capabilities with verifiable uncertainty for parts of interest to internal and external customers (fuel injectors, LIGA micro-gears, etc.)
- New measurement strategies provide best-in-world capabilities for challenging measurement tasks such as micro-pivot, reverse-taper microholes (fuel injectors) and knife-edge apertures (radiometric standards).
- 2× UMAP accuracy improvement
- New M48: error map of angular errors.

Future Plans
- Second M48 with several integrated microprobes.
- Standard Reference Material.

Micrometer level surface finish metrology



Setup detail & data for ballistics imaging designed experiment

Industry Needs Addressed
- Improved speed, resolution, and accuracy of surface finish measurements to enable improved productivity in U.S. manufacturing,
- traceability system for optical inspection devices of bullets and casings in crime laboratories according to recently developed guidelines of the American Society of Crime Laboratory Directors (ASCLD).

Impacts/Benefits
- NIST report cited heavily in National Academies' *Ballistic Imaging* casting doubt on usefulness of proposed database.
- Thousands of hits on Surface Metrology Algorithm Testing System (SMATS)
- Lots of praise from industry for random surface loaners

Objective
- Provide rapid, traceable optical 3D surface topography measurement at the micrometer level.

Technical Approach
- measure surfaces with optical techniques and compare with stylus methods,
- lead development of documentary standards for optical techniques,
- develop physical standards and measurement parameters for optical microscopes used in crime labs.

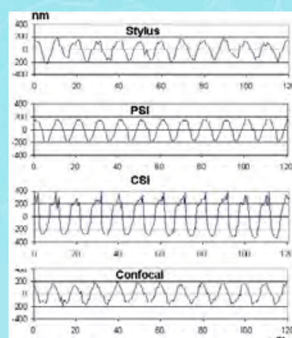
Highlights / Accomplishments
- Observation of large biases in roughness measurement by interferometric microscopes.
- Upgrading of SMATS.
- Leading ISO Project Team on Optical Methods, producing six draft standards.
- Acceptance and topography measurement of 150 units of SRM 2461, Standard Casings

Future Plans
- Investigate calibration standards and sources of error for optical methods.

A Few Highlights: Micrometer level surface finish metrology

Standard bullets completed

Standard casings distributed for testing



Coherence Scanning Interferom. shows a bias.

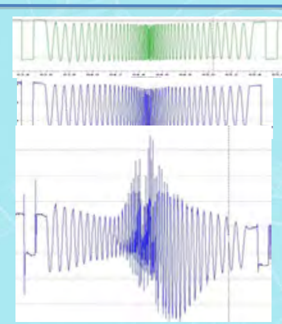
Stable Results for Indenter Tip Radius:
(196.83 ± 0.70) µm in 1996; Old Form Talysurf
(196.76 µm ± 0.72) µm in 2007; Demo Unit
(197.09 µm ± 0.64) µm in 2009; New Form Talysurf

After 2000 indents @ 981 N Force

Stylus

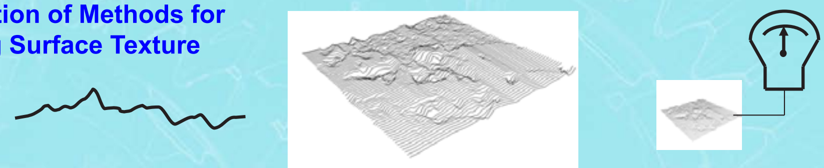
Confocal, 50X

Confocal, 10X



Measurement comparison on chirped sample

From ISO FDIS 25178-6, Classification of Methods for Measuring Surface Texture



Line Profiling,
Senses $Z(X)$

Contact Stylus Scanning,
Phase Shifting Interferometry,
Circular Interferometric Profiling,**
Optical Differential Profiling

Areal Topography,
Senses $Z(X,Y)$ or
$Z(X)$ as a function of Y*

Contact Stylus Scanning (FDIS),
Phase Shifting Interferometry (DIS),
Coherence Scanning Interferometry (DIS),
Confocal Microscopy (WD),
Confocal Chromatic Microscopy (FDIS),
Structured Light Projection,
Focus Variation Microscopy (CD),
Digital Holography Microscopy
Angle Resolved SEM,
SEM Stereoscopy,
Scanning Tunneling Microscopy†,
Atomic Force Microscopy†,
Optical Differential Profiling,
Point Autofocus Profiling (CD)

Area-Integrating

Total Integrated Scatter,
Angle Resolved Scatter,
Parallel Plate Capacitance,
Pneumatic

DIS – Draft International Standard

FDIS– Final DIS

CD – Committee Draft

WD – Working Draft

Nanomanufacturing Metrology Program

Enabling accuracy for nanotechnology manufacturing

Program Manager: Rick Silver

….makes accurate dimensional metrology critical to innovation and realization of quality products. Industries currently manufacturing at the nanoscale are challenged by the need for infrastructural metrology and standards to enable process control and to bring innovative new products to market. The goal is to advance U.S. leadership in nanomanufacturing through the development of physical standards traceable to the International System of Units….

Program Overview:

The Nanomanufacturing Metrology (NanoMet) Program focuses on the development of solutions to dimensional metrology needs of the nanomanufacturing industry by providing dimensional standards, calibrations and infrastructural metrology for measurements in the nanoscale having subnanometer precision.

- Semiconductor manufacturing, data storage, and photonics industries are currently the main customers
- The semiconductor manufacturing industry is a major current focus..
- Active interaction with industry leaders and industry consortia (such as SEMATECH); and strong participation in industrial roadmaps (e.g. International Technology Roadmap for Semiconductors).
- The goal is to provide the calibration techniques and artifacts which realize the *Système International d'unités (SI)* definition of the meter to meet the most demanding nanoscale industrial needs

Program Objectives:

- **Provide industry with accurate and timely dimensional scale metrology at the nanoscale to enhance U.S. productivity and innovation**
  - **Optical Linescale Metrology**
  - **Nanometer Scale Dimensional Metrology**
- **Provide accurate critical dimension (linewidth metrology) traceable to the meter.**
  - **Wafer Level AFM Metrology for Critical Dimension Measurements**
  - **Wafer Level SEM Metrology for Critical Dimension Measurements**
  - **Photomask Dimensional Metrology**
- **Provide accurate overlay and registration metrology with subnanometer accuracy to enable manufacturing of the most advanced, fastest semiconductor devices.**
  - **Overlay Instrument and Wafer Target Designs**

Major Programmatic Accomplishments

- Plenary presentation outlining new multi-technique nested uncertainties approach at the SPIE 2009 Metrology, Inspection, and Process Control Conference.
- First publication of procedures, methods, and uncertainty statement for traceable characterization of nanoscale sidewall angles.
- Implementation of first traceable wafer and mask level scanning electron microscope.
- Development of new Monte Carlo electron scattering model.
- Use of CD AFM to provide improved traceability and reference measurements for chrome on quartz photomask CDs.
- Implementation of first TEM-based tip width calibration for CD AFM.
- Commercial adoption of reversal methodology to identify field symmetry for improved overlay metrology.
- Demonstration of fully operational, in-house designed 193 nm excimer laser scatterfield optical microscope.

Nearly 40 publications in international journals including Optics Letters and Future Fab. Numerous invited and keynote presentations.

Linescale Metrology

Industry Needs Addressed
- Dimensional calibration of stage micrometers, linescales, meter bars, and 2D artifacts
- Traceability of SRMs
  (473, 475, 1692, 1960, 1961, 1965, 2059, 2800, 5000, 5001)

Impact/Benefit
- Golden standards for Semiconductor fabs
- Ten existing SRMs with more under development
- Top instrument in the Americas, Australia, and Asia

Objective
- Provide best-in-world 1D dimensional measurements to industry and NIST

Technical Approach
- Maintain and upgrade existing instrument
- Provide calibrations services to industry and support SRM development.
- Develop new instrument to replace existing world-leading but aging instrument

Highlights / Accomplishments
- LSI again places among top labs in Euromet.L-K7 international intercomparision.
- Proposal to develop new instrument accepted

Future Plans
- Improve electronics for existing instrument
- Continue to provide calibration services
- Develop new instrument
- Test frequency comb measurements of refractive index in LSI controlled environment

Nanometer Scale Dimensional Metrology: The Calibrated Atomic Force Microscope

Industry Needs Addressed
- Traceable nanoscale length measurements for the semiconductor and nanotechnology industries.
- Source of SI traceable scale calibration for SPM users – disseminated through commercial suppliers.

Impact/Benefit
- Traceable nanoscale length measurements and artifacts for US nanotechnology industries directly and through commercial standards suppliers and consortia.
- Strategic collaborations with industrial users of SPM to improve traceability and accuracy of industrial manufacturing metrology.

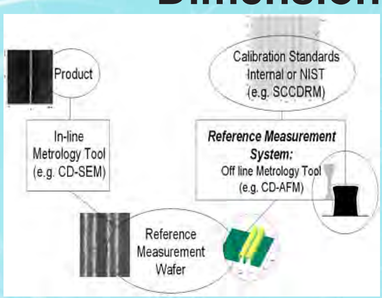
Objective
- Develop traceable SPM based dimensional measurement system by incorporating displacement interferometry with AFM to achieve traceability to the SI meter, for calibration of pitch, step height, and linewidth and disseminate this traceability internally and externally to NIST.

Technical Approach
- Integrate interferometric displacement metrology and SPM metrology platform to achieve best in class measurement performance.
- Collaborate with industrial users and SPM vendors to disseminate metrology practices.

Highlights / Accomplishments
- Traceable grating pitch measurement with 6×10^{-4} relative standard uncertainty in support of SEM metrology of Au nano particles for NIST RM project.
- Commenced tri-lateral comparison of pitch measurements with A-Star (Singapore) and a commercial standards supplier (ASM).

Future Plans
- Develop and characterize a new interferometrically traceable AFM – the T-AFM.
- Characterizations of Berkovich Indenters for possible development of nano-indentation standards.

Wafer Level AFM Metrology for Critical Dimension Measurements



Industry Needs Addressed
- Traceable nanoscale length measurements for the semiconductor industry.
- Traceable calibration samples for introducing *SI* traceability to a wide range of production relevant measurements.
- Support for measurement procedures development, and instrument evaluation.

Impact/Benefit
- Strategic collaboration with industry on development projects that leverage NIST expertise and introduce traceability to key measurements.
- Traceable nanoscale length measurements and artifacts to US semiconductor industry directly and through SEMATECH.

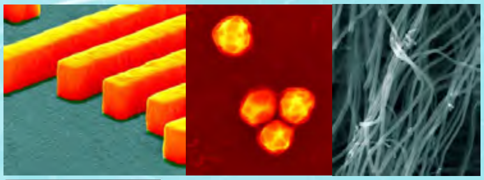
Objective
- *Develop traceable SPM based dimensional measurement systems for calibration of linewidth, step height, pitch, and related features, and disseminate the measurements and underlying technology to industry.*

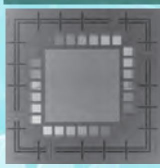
Technical Approach
- Introduce reference metrology techniques to production relevant measurements.
- Use methods such as ADF-TEM with aberration correction to introduce traceability to width measurements.
- Develop characterization and evaluation methods for key industry measurements such a sidewall angle, line edge roughness, and contact holes.

Highlights / Accomplishments
- First publication of procedures and methods for traceable characterization of nanoscale sidewall angle.
- Reduction of uncertainties for photomask length metrology.
- Completed measurements for NMI line width comparison.
- Evaluated and characterized new SEMATECH CD-AFM.
- Completed measurements and developed new procedures for ADF-TEM based width calibration.
- Measurements for photomask bilateral comparison with PTB

Future Plans
- Characterize and bring into operation a new CD-AFM.
- Develop lattice based lateral standards for CD-AFM.
- Incorporate an interferometry based drift monitoring system.
- Develop accurate measurement protocols and an uncertainty statement for prototype polysilicon linewidth standards.
- Explore the use of CD-AFMs for MEMS metrology

SEM Dimensional Metrology





From integrated circuits to nanomaterials scanning electron microscopy provides high-resolution images and accurate metrology solutions for manufacturing

Industry Needs Addressed
- Nano-scale dimensional metrology for semiconductor and nano-material manufacturing

Impact/Benefit
- Develop relevant sample preparation and characterization methods for scanning electron and ion microscopy-based metrology for industrial research, development and production use
- Develop and implement novel methods leading to more accurate and repeatable sound dimensional metrology solutions for the nano-meter scale manufacturing (1 nm = $1B in IC industry)

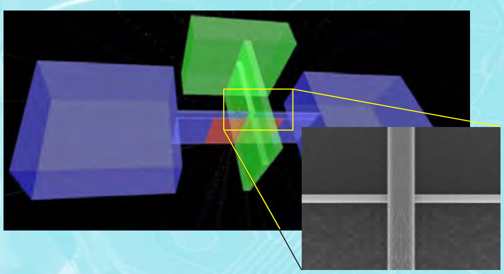
Objective
- Develop and validate measurement methods for the formulation and manufacture nanoparticles of interest in electronics- and nano-industry and nanomedicine.

Technical Approach
- Scanning electron microscopy
- Scanning helium ion microscopy
- Advanced laser interferometry and sophisticated measurement methods

Highlights / Accomplishments
- Developed two Reference Metrology SEMs equipped with laser interferometry, ready for traceable calibrations of masks, wafers and other samples.
- Characterized all important SEM parameters to minimize measurement uncertainty.
- Developed quantitative nanoparticle size analysis and dimensional metrology methods.
- Delivered four Reference Materials useful for a wide variety SEM, AFM and optical microscopy applications.

Future Plans
- Improve the accuracy of SEM-based dimensional metrology and help its rapid industrial introduction.

Wafer-Level SEM Metrology: SEM Modeling



Model FinFET and simulated image

Industry Needs Addressed
- SEM dimensional metrology (width and roughness) with <0.5 nm uncertainty.
- Instrument optimization and measurement limits assessment for defect detection.
- Error assessment for contour metrology.

Impact/Benefit
- Our modeling codes have been used at 5 instrument suppliers, 5 semiconductor electronics manufacturers, and >7 universities/research institutes .
- We alerted CD-SEM suppliers that their LER estimators were biased. Today all major suppliers claim bias-corrected metrics.

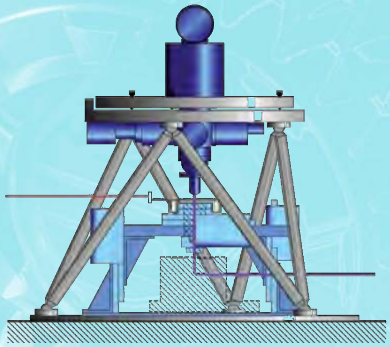
Objective
- Develop and validate instrument function models; use them to improve accuracy and define measurement best practices.

Technical Approach
- Develop sound physics-based models of electron transport and secondary electron generation.
- Use those models for Monte Carlo image simulation.
- Invert the models via Model-Based Library techniques to accurately determine geometry from images.

Highlights / Accomplishments
- Won a 2-year SEMATECH contract to develop SEM model for metrology and defect inspection.
- Gathered energy loss function curves for 57 materials.
- Increased repertoire of materials we can model with our most detailed model from 2 to 14 .
- Implemented new models for insulators (resist, SiO_2), special detector configurations…
- Studied and published sensitivity of metrology errors to choice of model.

Future Plans
- Model charging in insulators.
- Deliver modeling software to SEMATECH.

Photomask Dimensional metrology



Industry Needs Addressed
- Accurate metrology of integrated circuit photomasks

Impact/Benefit
- Accurate mask metrology is essential to maintaining the progressively lower cost/function embodied in Moore's Law.
- Accurate feature sizes and feature placement are required for lithography process robustness, leading to high yields of advanced semiconductor products.

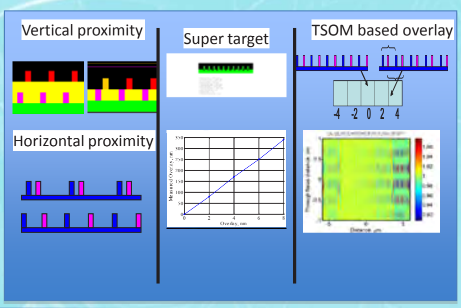
Objective
- Develop and validate measurement methods for the dimensions and placement of features on integrated circuit photomasks.

Technical Approach
- Produce Photomask Linewidth Standard Reference Materials
- Improve the NIST UV Scanning Microscope for photomask measurements.
- Improve the optical microscope image modeling needed for traceable optical dimensional metrology
- Develop rigorous analysis of parametric and random measurement uncertainty components
- Lead and participate in international comparisons of nanoscale linewidth measurement.

Highlights / Accomplishments
- UV Microscope is nearly operational after move and extensive update
- Parametric uncertainties for isolated lines and spaces on photomasks are being evaluated
  - over several parameter dimensions and
  - 400:1 range of linewidths
  - using two very different optical image models
  - and with both threshold and image library approaches

Future Plans
- Complete the parametric uncertainty study
- Collaborate on future photomask linewidth SRMs, including machine vision features as requested by customers

Overlay instrument and wafer target designs



Industry Needs Addressed
- Advanced overlay and registration metrology
- Dense targets that emulate device dimensions
- High throughput high-resolution process control methods
- Reduced overlay target sizes – industry identified critical real estate saving techniques using in-chip targets
- Overlay metrology for double patterning – very challenging

Impact/Benefit
- Enable double patterning to extend 193 nm lithography
- Helping metrology tool vendors develop new hardware and algorithms to extend optical overlay methods
- Creating new target designs and measurement techniques to enable sub-wavelength optical measurements

Objective
- Provide accurate overlay and registration metrology with subnanometer accuracy to enable manufacturing of the most advanced, fastest semiconductor devices.

Technical Approach
- In collaboration with SEMATECH – design and have fabricated new targets and test wafers
- Construct/ modify optical tools with custom illumination optics and develop new data acquisition methods
- Extensive modeling using electromagnetic simulations tools to optimize parameters and fitting procedures
- Use experimental targets and test wafers to validate models and implement quantitative optical measurement procedures with sub-nanometer sensitivity and accuracy

Highlights / Accomplishments
- Adopted by leading semiconductor manufacturers and metrology tool developers
- Patented several overlay target designs
- Supertarget design – magnify overlay with design rule targets
- New results demonstrating importance and necessity of horizontal and vertical optical interactions
- TSOM based overlay targets: extreme sensitivity and robust to process variations and optical aberrations
- Performed overlay measurements with the new 193 nm tool
- Identified important challenges and limitations in industry funded scatterometry-based overlay study.

Future Plans
- Experimentally evaluate recent design rule target designs showing good overlay sensitivity based on simulations
- Further reduce overall overlay target sizes

Next-Generation Nanometrology Program (Next-Gen Program)

Solving industry needs of tomorrow …

John Kramar
Program Manager

Precision Engineering Division Program Review
September 22, 2009

Next-Gen Program

Develop measurement science and technology approaches to address the measurement challenges of next-generation nanofabrication.

Challenges:

- New manufacturing processes
  - self assembly
  - bio-manufacturing
  - massively parallel atomically precise manufacturing
- New measurands
  - nanoparticles (size and shape/form)
  - nanotubes and nanowires
  - photonic crystals, metamaterials
  - bio materials, soft objects
- Higher resolution
  - atomic resolution
- Higher relative precision
- Higher throughput

Next-Gen Program

Develop measurement science and technology approaches to address the measurement challenges of next-generation nanofabrication

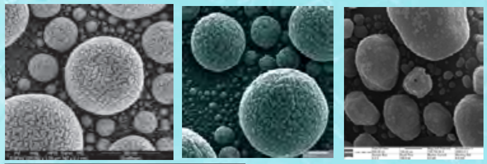
Approaches:

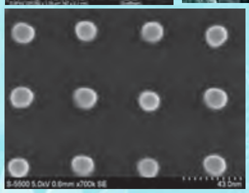
- New metrology techniques
  - helium ion microscopy
  - scatterfield microscopy
  - 193 nm optical microscope
- Innovations and enhancements in existing methods
  - pixel sorting and correlation averaging for noise reduction
  - quantifiable and repeatable performance metrics
- Intrinsic standards, natural phenomena based (*not direct realizations of the definition of the meter*)
  - atom-based standards (pitch, height, width)
- Sampling protocols and sample preparation techniques
  - non-distorting substrate attachment
  - soft material imaging technique innovations
  - trapping and manipulating individual nanoparticles for measuring

Next-Gen Program: Projects

- Scanning Particle-Beam Microscope Innovations
  Andras Vladar
- Atomic Force Microsope (AFM) Nanoparticle Metrology
  John Dagata
- Nanoparticle Manipulation Metrology
  Tom LeBrun
- Atom-Based Standards and Fabrication
  - Molecular Measuring Machine
    John Kramar
  - UHV-STM Nanofabrication and Metrology
    Rick Silver
  - Atom-based Step Height Standards
    Ron Dixson
- High-Throughput Nanometrology with Scatterfield Microscopy
  Rick Silver

Scanning Particle-Beam Microscope Innovations





Scanning electron and ion microscopy is indispensable for very high-resolution imaging, accurate dimensional metrology, nano-milling and nano-lithography, down to the few nanometer scale.

Industry Needs Addressed
- High-resolution nano-scale imaging, dimensional metrology methods and fabrication for semiconductor and nano-material research and development

Impact/Benefit
- Expanding the limits of scanning electron and ion microscopy
- Reliable sub-nanometer resolution imaging and metrology solutions and methods for scanning electron and ion microscopy-based metrology use in research and development
- Develop and implement novel three-dimensional metrology methods in the 10 mm to 10 nm scale

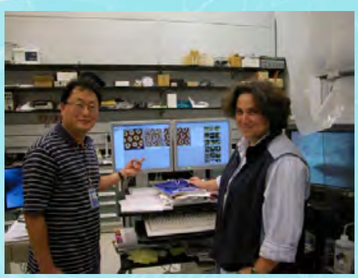
Objective
- Develop best-in-the-world scanning electron and helium ion microscopy that is capable of unprecedented quality in imaging and measurements for future electronics- and nano-industry and nanomedicine.

Technical Approach
- Scanning electron microscopy
- Scanning helium ion microscopy
- Advanced laser interferometry and sophisticated measurement methods

Highlights / Accomplishments
- Cooperation in the implementation of He ion scanning microscopy (Zeiss)
- Developed sophisticated new image collection and measurement methods.
- Developed He ion lithography and nanomilling methods with close to 10 nm smallest fabricated structures.

Future Plans
- Implementation of accurate 3D dimensional metrology and nanometer scale fabrication methods.

AFM nanoparticle metrology



Atomic Force Microscope Nanoparticle Metrology project guest researchers HyeongGon Kang and Natalia Farkas discussing atomic force microscope images of erythrocytes.

Industry Needs Addressed
- Nanoparticle metrology for drug discovery and pharmaceutical manufacturing

Impact/Benefit
- This work has developed novel methods for attaching targeted nanoparticle delivery systems to surfaces, leading to more efficient physical optimization of drug formulations. Regulatory approval of NDS formulations by the US Food & Drug Administration is growing. However, standardized measurement protocols for ensuring manufacturability and potency of NDS formulations do not yet exist, thus slowing approval of these promising therapeutic and diagnostic for clinical trials and their availability to patients who could benefit from them.

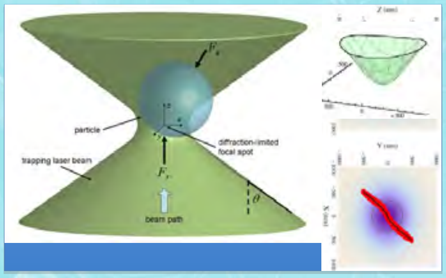
Objective
- Develop and validate measurement methods for the formulation and manufacture nanoparticles of interest in healthcare, pharmaceutical manufacturing, and nanomedicine.

Technical Approach
- Scanning probe microscopy.
- Dynamic light scattering and zeta potential.
- Optical microscopy.

Highlights / Accomplishments
- Developed immobilization methods for liposome-based targeted nanoparticle delivery systems.
- Characterized super-paramagnetic iron-oxide nanoparticles for magnetic resonance imaging contrast agents.
- Investigated quantitative nanoparticle size analysis by scanning probe and dynamic light scattering methods.
- Demonstrated optimized attachment of red blood cells to surfaces for combined optical and scanning probe microscopy.

Future Plans
- Investigate the correlation of mechanical and chemical responses of cell membrane due to protein expression.

Nanoparticle Manipulation Metrology



Industry Needs Addressed
- Nanoparticle manipulation for sorting and testing
- Fabrication of nanoparticle-based devices
- Prototype and test of nanodevices

Impact/Benefit
- Available nanomanipulation capabilities are rudimentary and inefficient — when they exist
- Allows sampling of nanoparticle materials based on desired characteristics, or rare events
- Allows probing biological systems with reduced danger of optical damage

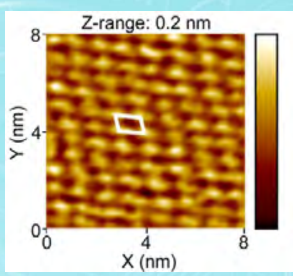
Objective
- Demonstrate controlled optical trapping to enable the accurate manipulation and positioning of nanoparticles

Technical Approach
- Simulate controlled trapping to allow test under practical conditions of lab and controller
- Measure full trapping force profile in laboratory to allow accurate predictions by simulation
- Demonstrate controlled trapping in lab based on measurements and simulation

Highlights / Accomplishments
- Measured full nonlinear force profiles
- Simulations demonstrate feasibility of new method

Future Plans
- Improving positioning, force measurement, speed of simulations, and bandwidth
- Developing proposal for last year of thesis at UMD
- Lab experiment to demonstrate controlled trapping this year

Molecular Measuring Machine



Closed-loop, servo-controlled, nanometer resolution imaging and metrology of a molecular crystal, ((TEET)[Ni(dmit)$_2$]$_2$. a = (1.09 ± 0.07) nm, b = (0.70 ± 0.05) nm, γ = 106° ± 3°

Industry Needs Addressed
- Traceability path for nanometer-accurate, two-dimensional position metrology of nanometer-scale objects and features over macroscopic areas

Impact/Benefit
- Validates the use of specific crystals or natural objects as intrinsic reference standards
- Atomic-accuracy over real-world distances
- Provided SI traceability for NASA's Chandra X-ray Observatory spectroscopic measurements by measuring HETG and LETG grating standards (200 nm and 400 nm pitch)

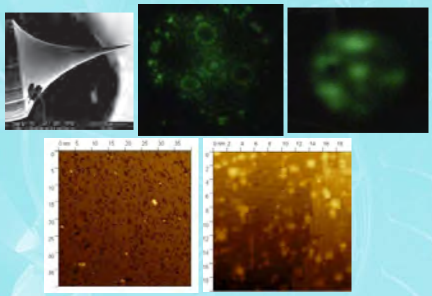
Objective
- Develop a measuring instrument for traceable nanometer-resolution and nanometer-accurate measurements over a 50 mm by 50 mm area

Technical Approach
- Scanning probe microscopy
- Precision motion stages
- Heterodyne interferometry
- Vacuum, temperature controlled environment
- Interferometer-controlled, closed-loop scanning
- Probe-based lithography used for artifact creation

Highlights / Accomplishments
- Developed a compact, compound actuator and sensor for controlled metrological motion and measurement
- Developed a method to perform high resolution heterodyne interferometry in vacuum using fiber optic beam delivery

Future Plans
- Validate the use of a crystal lattice as a reference artifact by imaging and measuring lattice constants of a perfect crystal over a 1 um by 1 um area

Atom-based standards and fabrication



Industry Needs Addressed
- Calibration of a prototype atom-based 1 nm step height standard with an accuracy of 10 pm.
- Fabrication of structures smaller than 5 nm in dimension for use in standards and nanotechnology research.

Impact/Benefit
- Development of both the metrology for atomic resolution measurements and the ability to fabricate and measure with atomic precision on the nanometer scale
- Industrial round robin comparison of atom-based 1 nm step height standards to validate the accuracy of the method and of the industry metrology base.

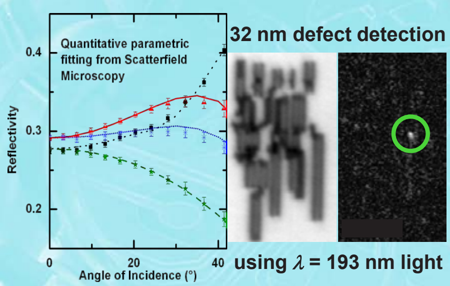
Objective
- Develop intrinsic calibration standards based on the crystalline lattice

Technical Approach
- Develop atomically ordered silicon (100) surfaces using wet chemical etching and high temperature annealing
- Develop methods for repeatably producing atomically-sharp tungsten and alternative-material tips, and evaluate their atomic resolution imaging capabilities
- Develop improved nanofabrication methods using the STM to write atomic-scale features on hydrogen-terminated silicon substrates using scanning probe oxidation for accurate nano-standard development.

Highlights / Accomplishments
- Awarded a 5 years, 3 phase DARPA contract to conduct collaborative research in atomically precise positioning, patterning and metrology.
- Repeatable silicon processing using high temperature annealing process achieved
- Preparation and in-situ characterization of STM tips to obtain atomic resolution STM Images
- Fiducial mark design and reticle fabrication completed

Future Plans
- Optimization of the writing and de-passivation methods
- Test wafer die in UHV high temperature Si process

High-throughput nanometrology with Scatterfield microscopy



Industry Needs Addressed
- Decreasing critical dimensions must be rapidly measured for process control during manufacturing
- As dimensions decrease, the size of defect which would adversely affect manufacturing yield also decreases
- High-resolution, high-throughput metrology solutions are needed throughout nanoscale manufacturing.

Impact/Benefit
- Reduced cost process control solutions using high-throughput, non-contact optic techniques
- Defect detection is made over large fields-of-view, allowing quick corrective measures for higher yields.
- Multi-nested analysis permits lower uncertainties by combining optics with other metrology tools.

Objective
- Develop optical methods with extremely high throughput rates to accurately measure nanoscale features and objects

Technical Approach
- Scatterfield microscopy, combining the best elements of scatterometry and optical microscopy
- Engineering of the illumination at the sample to permit a single angle of incidence, or specific angles which would enhance sensitivity to a particular feature
- Advancing the state-of-the-art in optical microscopy by coupling these new techniques with a shorter wavelength, λ = 193 nm, for optimal performance

Highlights / Accomplishments
- Construction of deep ultraviolet (λ = 193 nm) and visible (λ = 450 nm to 750 nm) scatterfield platforms
- Quantitative sub-wavelength dimensional metrology
- Exhaustive experimental and simulation studies for defect detection

Future Plans
- Sub-30 nm defect analysis on intentional defect arrays provided by SEMATECH
- Quantitative measurements of arrayed targets using angle-resolved and wavelength-resolved visible light
- Robust development of sampling strategies to reduce uncertainties by combining optical microscopy with other established technologies.